Abstracts.

Arranged by W. H. Thomas, A.R.S.M.

Abstractors.—S. F. Birch, Ph.D., D.I.C., S. W. Cooke, B.Sc., P. Docksey, B.A., H. S. Garlick, B.Sc., A.I.C., W. N. Hoyte, B.A., H. G. Shatwell, Ph.D., M.Sc., E. R. B. Skipwith, B.A., A. R. Stark, B.Sc., A.I.C., W. H. Thomas, A.R.S.M., T. Wiskin.

General.

1. Baku. Chaildag Petroleum. *Oil Bulletin*, 1927, **22**, 10.

A second rich oil bearing strata has been opened up at Chaildag, 60 versts from Baku. The oil is the better grade benzine with S.G. 0·820 and in quality excels the light Bibi-Eibat petroleum. Three wells are drilling, one of which has entered the Maikop layers, hitherto not exploited on the Apsheron peninsular. T. W.

2. Binagadi Oil Beds. *Oil Bulletin*, 1927, **22**, 10.

A continuation of the Binagadi Oil beds, in the so-called Kirmakinsky layers, has been discovered to the west of Binagadi. At the present time a well 324 m. deep is in exploitation, yielding a high quality oil. T. W.

3. New Oil Moorings in Batoum. *Oil Bulletin*, 1927, **23**, 23.

Owing to the mole now being lengthened, 2 more oil loading moorings are available since October. The new moorings permit deep seated ocean going tankers to tie up closely, and will curtail overhead charges, diminish freights, increase the numbers of voyages, as well as creating advantageous conditions for the exporting abroad of oil and its products.

The capacity of the port has risen already by 20 per cent. T. W.

4. Tankers Built in France. *Oil Bulletin*, 1927, **23**, 23.

The 2 tankers ordered in France in 1926 will be the first in S.S.S.R. of the capacity—10 thous. tons. The first is to be completed in May, 1928, and the second some months later. T. W.

5. Baku-Batoum Pipe-Line and Refineries in Batoum. Report by by B. Y. Starodoub. *Oil Bulletin*, 1927, **23**, 11.

The route for the pipe-line is Baku-Sumgait, over the pass Marasy, Karayazvy-Evlach, thence parallel with the railway to Batoum. This route was chosen as being the shortest in conformity with natural conditions, and for economical considerations a straight line from Baku to Batoum is about 700 klm. and the length of the pipe-line will be 842 klm. with 10 in. pipes. It is estimated that 1·5 mill. tons of Balachani oil will be pumped through this 10 in. line, and through the existing 8 in. line 820 thous. tons of Surachani oil. 14 relieving stations are projected—2 less than on the existing pipe-line. This is owing to the shortening of the route by 42 klm. and the more equidistant spacing of the stations, the average distance being about 60 klm.,

and only in 2 districts 120 klm. where self gravitation takes place from the mountain passes. These two passes Marasy and Suramsky are 800 m. above sea level. Two variations have been surveyed for the Suramsky pass—a tunnel and round by the old railroad track.

Three 300 h.p. installations are to be erected, two working, one in reserve. In the event of connecting the 8 in. with the 10 in. pipe-line an additional 300 h.p. installation will be installed.

The pipes will be welded together by the acetylene process, this joint being superior to the screwed connection, giving a more dependable joint, a quicker laying of the line and a cheaper pipe. The line, buried 1 ft. in the ground, passes over 10 large railway bridges and over small ravines and streams on special supports.

The refining plant has been calculated to distill 820 thous. tons Surachani oil with one battery, and 1,480 thous. tons light Balachani oil in two batteries, these batteries working on the regenerator system. 200 hectares have been allotted for the erection of the refineries between the rivers Korolis-Tskhali and Kuba-Tskhali, 2½ klm. from Batoum, with provision for further extension. The crude oil storage capacity is projected to be constructed across the Korolis-Tskhali, and that for finished products across the Kuba-Tskhali so that on the refinery territory there will be no reservoirs. The outlay of the refineries is such that the products will move in one direction without cross-pumping and with the least length of piping, the crude oil gravitating into the kerosine battery. The power to be installed is 3000 klw. with 3 turbine alternators. Water is supplied through a 600 m.m. wooden flume by gravitation from a pumping plant, 4·5 klm. above the refinery, on the river Korolis-Tskhali. All other erections (shops, stores, offices) are situated outside the works territory near to the railway.

The estimated output is as follows: Benzine 287 thousand tons, ligroin 24 thousand tons, kerosine 740 thousand tons, gas oil 220 thousand tons, transformer lubricants 295 thousand tons, other lubricating oils 394 thousand tons, asphalt 115 thousand tons, fuel and loss 377 thousand tons.

The cost of the pipeline is estimated at 32 million rbls., the pipes costing 14,750 thousand rbls. and pumping stations 10 million rbls. The estimate for erection of refineries is 21·2 million rbls. Together, with the reconstruction of the kerosine pipeline (3·5 million rbls.) and the erection of a workman's settlement in Batoum, both undertakings will cost 60 million rbls.

During the first year 232 klm. of pipeline will be laid from Batoum to Khashturi, with probably an additional 196 klm. to Poily. There will be 3 pumping stations on the Batoum-Poily section. The refinery installations have already been commenced and the kerosine battery should be completed by the 1st October, 1928, and the kerosine-lubricant battery during the first or second quarter of 1928/29. The whole construction is to be completed in 5 years.

The cost of pumping through the pipeline is calculated at 5·75 cop. per pood, while railway transport is 16 cop. and the N.K.P.C. charges 7·32 cop. for delivering kerosine through the kerosine pipeline. T. W.

6. The Swelling of Bentonite and its Control. C. W. Davis. *Ind. and Eng. Chem.*, 1927, **19**, 12, 1350.

In drilling wells in search for oil, potash, or other minerals, water is generally used. If bentonite is encountered the drilling water causes the bentonite to swell and form a sticky mass which flows into the hole. As more water becomes mixed in, the mass becomes less viscous and finally results in a permanent suspension. There is a continuous transition from the stiffest paste to the most mobile suspension, all dilutions being capable of holding

other minerals in suspension. If oil is present, bentonite promotes the formation of water-oil emulsions, which may be very viscous or practically solid. In any case bailing is very difficult and tedious. The sides of the hole may swell, heave, and cave, and may result in casing collapse. It is therefore evident that the prevention of the swelling of bentonite encountered in drilling is an important problem.

An understanding of the swelling of crude bentonite is complicated by the fact that the material is not a pure substance; that it has no definite chemical composition; that its composition may change in the presence of salt solutions; and that it frequently contains varying amounts of soluble salts. The addition of acid or alkali decreases the swelling power of bentonite in water, but this effect is very small in the range pH 4 to 9. The measurable effect of neutral salts on swelling seems to depend somewhat on the concentration. Results of experiments indicate that each neutral sulphate is less effective than its corresponding chloride and show a slight difference in the effects of neutral cations on the swelling of bentonite. However, the differences are so small as to preclude a statement on the specificity of the ions.

Material whose apparent expansion in water is due to breaking down should cause trouble in wet drilling only through caving of the well walls, since the increase in volume is small and the resulting product is not pasty, but certain bentonites swell markedly in water and are known to cause difficulties. The tests described in the paper lead to the conclusions that lubricating oils, kerosene or gasoline, could be used to replace the water in wet drilling through bentonites to prevent swelling and breaking down. However, water would have to be excluded to prevent the formation of emulsions. Cheap salts could be used to form dilute solutions that would cut down the swelling effect of bentonites in water considerably, but they would probably not prevent caving. The addition of a greater amount of these salts would apparently prevent all detrimental action of the solutions on bentonite. For some bentonites, sodium chloride evidently gives satisfactory results; other bentonites, however, become soft in a saturated solution of this salt. It seems possible that these bentonites may remain firm in a solution of some other common salt, so that drilling difficulties caused by bentonite may be overcome in all cases. S. W. C.

7. Florida Oil Possibilities. E. A. Hill. *Oil Age*, 1927, 24, **11**, 38, 53—54.

After upward of 20 years of desultory and sporadic drilling Florida is again in the throes of an exploratory campaign directed towards oil discovery. Although some tests have been drilled with proper regard for the findings of responsible geologists yet no successful well has so far been completed. The geologic problem is examined with a view of ascertaining somewhat of its complex aspects.

It would seem paleogeographically that Florida except the extreme southern tip was submerged largely throughout the Paleozoic era and conditions are somewhat like those of the oil and gas fields of Pennsylvania and adjoining states of Illinois, of Oklahoma and Kansas, and of portions of Texas. During the later epochs of the Pennsylvanian, throughout the Permian period and in the opening periods of the Mesozoic era, Florida was dry land, and subject to erosional processes. Universal submergence occurred during the middle Cretaceous, and emergence from the sea during the epi-Mesozoic interval of the Upper Cretaceous, followed by erosion. With the advent of Tertiary times Florida was again submerged. Thus the region was under water for long periods; it was out of the water and denudation in progress at other times; then it was again submerged by the seas, and again emerged.

If possible, oil in Florida had genesis in the sedimentary beds of the Palæozoics, earth movements have been rightly timed and of adequate duration, and sufficiently extensive in scope to have provided for its gathering by migration to catchment reservoirs in the Cretaceous rocks, which have a thickness of some 3,000 feet or more.

It is not impossible that migration could have found ingress and accumulation under favourable conditions of porosity and structure in Tertiary strata; but oil is not believed to be likely of discovery in formations of that period in the State. The Tertiary system of rocks has a thickness of somewhat near 2,300 feet in a large part of north western Florida.

Earth movements disturbed all strata antecedent to the Pleistocene in the Florida province. The sediments bear ample testimony to the fact of at least a reasonable degree of organic material. Some actual crude oil under normal earth conditions has been obtained from wells drilled in the State and actual probabilities of commercial supplies seem to be indicated: but it is extremely difficult, often almost impossible, to determine if structural conditions are present or not.

Depths to possible horizons of saturation if actually present will not be found excessive as drilling practice is understood to-day. Time, money, experienced drillers working under the counsel of competent geologists, adequate modern drilling equipment of combination cable and rotary types only, will determine the presence of oil in paying amount in the State.

H. S. G.

8. Electricity in the Petroleum Industry. R. H. Rogers. Oil and Gas J. Vol. 26. No. 34.

A two-fold bond exists between electric power and the oil fields. The electrical industry absorbs many products of the petroleum industry: fuel oil, transformer oil, lubricating oil, carbon in various forms and paraffin wax. At the same time, although the petroleum industry is the least electrified of the great concerns, there are certain advantages to be obtained from the use of electric power.

Electrification makes it possible to use fuel cheaply in large central power stations. The electric motors then used as power units are far more easily handled, and require less preparation in the way of foundations, etc., than other power units.

If a refinery runs its own generating plant there is the advantage that process steam can be obtained at any desired pressure by "bleeding" from the appropriate stage of the turbine. On the other hand, obtaining the supply from a central station and transforming down to the correct voltage at the refinery has the great advantage that failure of the power supply is less likely to occur.

For drilling, both with cable and rotary tools, electric motors are extensively used. 50 H.P. motors at 720-900 r.p.m. are used for cable drilling, and these may be run in such a way as to take full advantage of the elasticity of the cable. For hoisting work the motor can be speeded up and run at constant speed.

For rotary drilling motors of slightly higher horse-power may be used with advantage. The slush pumps are driven by two speed motors at the lowest speed. The higher speed is used for pulling rods and tubing and bailing. All this equipment can be obtained in a vapour protected form.

Electrically-driven compressors for air-lift pumps are very successful, as also are electrically-operated contrifugal pumps for pumping water and oil. The application of motors to serve as water and oil circulators and

pumps in natural gasoline plants and refineries is too wide to be given detailed description in this paper. P. D.

9. Manufacture of Highly Active Carbon Blocks or Granules. Chemische Werke Carbon Gesellschaft. E.P. 266,674. Nov. 29, 1927. Conv. Feb. 24, 1926.

The patent describes a process for the manufacture of a hard moulded, uniformly active carbon, in which finely divided non-active carbon or carbonisable material is homogeneously mixed with an organic binding medium. The latter contains a proportion of a soluble inorganic substance. The mixture is moulded under pressure, dried, carbonised, calcined and activated in a known manner by means of gases or vapours in order to produce hard blocks or granules.

H. S. G.

10. A Method of Preparing Artificial Petroleum and Decolourising Carbonaceous Substances from Rubber Scrap or Vulcanised Rubber Waste. Nishida and Shimada. E.P. 282,565. Dec. 29th, 1927.

Vulcanised rubber waste is crushed and mixed with twice to four times its weight of either Japanese acid earth, fullers or Florida earth and distilled. Decomposition occurs below 100° C. and oil and water vapour distill from 100° C.—350° C. The oil is fractionated and washed with dilute sulphuric acid, soda and water to give gasoline, kerosine and neutral heavy oils, while the residue is a black hard material suitable as a decolourising earth. The distillate amounts to 60 per cent. of the charged rubber.

Otherwise the rubber waste is distilled alone and the distillate dropped on to heated earth in a retort, the subsequent steps being as detailed above.

A. R. S.

11. Improvements Relating to the Treatment of Acid Sludge or Acid Resin Obtained from the Refining of Mineral Oils. J. Pickering. E.P. 279,613. Nov. 3, 1927.

The invention provides for a process for the treatment of acid sludge for the purpose of removing sulphuric acid from residues obtained when treating mineral oils with sulphuric acid for the purpose of obtaining a neutral sludge. The sludge whilst being stirred and heated to a temperature below the boiling point of an organic solvent (preferably acetone), capable of mixing with water or an aqueous solution and sulphuric acid, has the solvent sprayed thereon until the sludge is completely dissolved, the quantity of solvent added depending upon the consistency and nature of the acid sludge treated. The dissolved sludge is then treated with water, or an aqueous solution of a suitable salt (sodium chloride or carbonate) capable of being mixed with the organic solvent in any proportions, in which the sludge is not directly soluble and which is added in quantities in accordance with the nature of the sludge and the chemical and physical condition thereof. This is followed by one or more washings with water or an aqueous solution for the purpose of removing remaining traces of solvent and acid from the sludge. The washing solution may be re-used in one or more of the subsequent washing operations whereby it is enriched with solvent and can then be either concentrated or be used for dissolving fresh charges of acid sludge.

S. W. C.

See also Abstracts Nos. 21, 34, 36, 40, 52, 71 and 84.

Chemistry of Petroleum.

12. Synthesis of Methane from Water Gas. Hightower and White. *Ind. Eng. Chem.* 20, 1, 10.

The apparatus consists of a pyrex tube containing the catalyst in an electric furnace, the temperature being observed by means of thermocouples. The catalyst was nickel supported on unglazed porcelain, the proportion of nickel in the finished catalyst being between 1·3 per cent–4·9 per cent., the starting compound being nickel nitrate. The gas analyses were absorption for carbon monoxide, carbon dioxide, unsaturated hydrocarbons and oxygen, while methane was done by explosion.

The decomposition of CO, according to the equation—

$$2CO = C + CO_2,$$

was somewhat erratic, although appearing to a small extent between 300-370° C. It appears to happen to a smaller extent with new catalysts, and therefore may be autocatalytic.

The path of the reactions—

$$CO + 3H_2 = CH_4 + H_2O$$
$$CO_2 + 4H_2 = CH_4 + 2H_2O$$
$$2CO + 2H_2 = CH_4 + CO_2$$

depend on the following :—(1) is repressed by the presence of water vapour and predominates if the water vapour is regularly removed. Reaction (2) appears to be uncomplicated ; while (3) occurs in presence of much water vapour.

For the reactions—

$$CO + H_2O = CO_2 + H_2.$$
$$C + 2H_2 = CH_4.$$
$$2CO = C + CO_2$$

the equilibrium constants at 350° C. have been found. Thus $\log_e K$ for the combined reactions appears to be in the neighbourhood of 19·6.

A. R. S.

13. The Transformation of Phenols into Hydrocarbons with Catalyzers and Compressed Hydrogen. Kling and Florentin. *Brenn. Chem.* 8, 20, 331.

All aromatic and hydroaromatic hydroxyl compounds form saturated hydrocarbons in good yield when heated under pressure with hydrogen and a dehydrogenating catalyst such as aluminium clay, alumina, silicic acid, etc. At 480°, phenol with 5 per cent. of aluminium and hydrogen under pressure of 70–80 kg. per sq. cm. was acted upon rapidly, and in 4 hours 40 per cent. of it was converted into light hydrocarbons, sp. gr. 0·874, without noticeable formation of dephenyl oxide. Of this 13 per cent. boils at 70–79°, sp. gr. 0·855, and 72 per cent. at 79–83°, sp. gr. 0·875. The latter fraction has the character of benzol and contains small quantities of aliphatic hydrocarbons. The commercial mixture of the three cresols under the same conditions yielded over 35 per cent. of the cresols, which distil in part at 85 to 115°, sp. gr. 0·863. The $-CH_3$ group does not appear to be attacked. In the same way cyclohexanol was carried over almost quantitatively in 2 hours at 460–470 to light aliphatic hydrocarbons boiling at 70–90°. This reduction, which can be extended to complex commercial products like phenol oils, low-temperature tars, etc., requires a catalyzer of medium strength and sufficient hydrogen pressure in order to produce saturated hydrocarbons.

W. H. T.

14. Recent Asphalt Theories. Nellensteyn. *Zeitsche. angew. Chem.* 23, 30, 1195.

Asphalt is a highly protected carbon oleosol. It contains three principal constituents—the carbon particles, the protective substance, and the medium. The first two form the asphalt micells. The stability of the system is important for the use of the asphalt for street-building and depends upon the relations between micell and medium. If the micell is insoluble without itself being broken down, the so-called reversible flocking takes place, as when asphalt is determined with petroleum ether. This flocking depends almost altogether upon the surface tension between micell and medium. Liquids with a lower surface tension, like benzine and ether, are flocking agencies; those with higher, like benzol, are solvent. The critical surface tension for producing flocking is 24–25 degrees per cm. Carbon tetrachloride has this surface tension, and can therefore be used as stability reagent. If the micell itself is destroyed, irreversible flocking takes place, and the adsorption relation between the carbon particles and the protective substance is destroyed.

W. H. T.

15. Polymerisation of the Amylenes. James F. Norris and Jozua N. Joubert. *J.A.C.S.*, 1927, 49, 873–886.

The action of various concentrations of sulphuric acid at room temperature on the five amylenes and the chemical properties of the resulting diamylenes have been studied. In all experiments equimolecular quantities of the hydrocarbon and the acid were used. It was found that of the five amylenes, trimethylethylene and *unsym.* methylethylethylene were the most readily polymerised, whilst *iso* propylethylene was the only one to polymerise without first dissolving in the acid. Although trimethylethylene and *unsym.* methylethylethylene yield *tert.* amyl alcohol on dilution of the acid followed by saturation with ammonium sulphate, pentene–1 and pentene–2 only gave *sec.* amyl alcohol after dilution and hydrolysis proving that they were produced through the alkyl sulphuric acids. The formation of the diamylenes appears to be the result of the dehydration of the amyl alcohol in solution or of that formed by hydrolysis of the alkyl sulphuric acid.

The diamylenes were shown to be olefines much less reactive than the parent hydrocarbons; some light was thrown on their structure by the examination of the compounds obtained when the diamylene ozonides were decomposed in the usual manner. It was also found that the amylenes did not condense with primary or secondary alkyl halides when heated to 100° C. in the presence of lime or zinc oxide. Under similar conditions trimethylethylene condensed with tertiary halides but *iso* propyethylene did not. Improved methods for preparing the amylenes are given. S. F. B.

16. The Re-arrangement of *Iso* propylethylene to Trimethylethylene and the Pyrogenic Decomposition of Pentene–2 and Trimethylethylene. James F. Morris and Raymond Reuter. *J.A.C.S.*, 1927, 49, 2624–2640.

A detailed study of the properties of the amylenes is of particular interest on account of the fact that certain of these hydrocarbons are formed in the cracking of petroleum and are now of importance as sources from which amyl alcohols are prepared on a large scale. The results obtained by the authors earlier in the year (see previous abstract) showed that the chemical reactivity of the three amylenes pentene -2, trimethylethylene and *iso*-propylethylene with inorganic acids varies greatly with their structure. The present work indicates that there are similar differences in their behaviour when they are subjected to the action of heat. *Iso*-propylethylene when

passed over alumina at 450° re-arranges itself to trimethylethylene to the extent of 10 per cent. Under similar conditions aluminium sulphate produces 47 per cent. re-arrangement and phosphoric acid 29 per cent. The fact that this re-arrangement takes place under the influence of catalysts led the authors to study the effect of these catalysts at elevated temperatures on *iso*-amyl alcohol—the method generally employed to prepare "amylene." It was found that the isomerisation was then slightly greater than when the hydrocarbon itself was heated. The efficiency of the catalysts in producing the change was also in the same order. Under similar conditions no re-arrangement of the trimethylethylene to *iso*-propylethylene could be detected. Pentene-2 also remained unaffected and showed no tendency to form branched chain isomers. When heated to between 575° and 650° C. both pentene-2 and trimethylethylene are cracked and yield liquid and gaseous products which are quantitatively analysed as follows :—The gases were analysed for :—(1) Total unsaturation by means of bromine water. (2) Unsaturated hydrocarbons other than ethylene by means of 87 per cent sulphuric acid. (3) Methane by explosion with air after removal of unsaturated compounds. (4) Total unsaturated compounds by fractionation of the bromides obtained when the gas was treated at -20° to -10° with bromine dissolved in carbon tetrachloride and also by fractionation of the hydrogen chloride addition compounds ; the liquid condensate was analysed for (5) Compounds containing two radicals linked to an unsaturated carbon atom by treatment with 60 per cent. sulphuric acid. (6) Unsaturated compounds by fractionating (*a*) the bromides produced when the fraction boiling up to 34° was treated with bromine at -17° to -10° and (*b*) The chlorides produced by the addition of hydrogen chloride to the same fraction. The results obtained indicated the presence of methane, butylene, butadiene, propylene, ethylene and higher hydrocarbons. An interpretation of the mechanism of the decomposition based on these results is given. Although *iso*-propylethylene is stable towards dilute sulphuric acid, it was shown that it can be made to re-arrange to trimethylethylene to a small extent by the action of acid of such concentration that polymerisation does not take place. The boiling-point, melting-point, refractive index and densities at various temperatures of trimethylethylene, pentene -2, *iso*-propylethylene and *tertiary* amyl alcohol have been redetermined on carefully purified materials. S. F. B.

17. Lead Sulphide Treating in Refining. G. L. Rowsey and J. F. Whitehurst. *Oil & Gas. J.*, 927, 26, 32.

An account is given of the difficulties encountered when the Amarillo Refining Co., Texas, commenced operating on Panhandle crude oil. Apart from the excessive corrosion of plant caused by the highly sulphurous distillates, as much as 1·2 lb. of litharge per barrel were required for sweetening, which necessitated air blowing for 16–18 hours. The treating losses were often as high as 7 per cent. By using a preliminary soda wash to remove hydrogen sulphide and reduce the mercaptan content, it was found that the litharge treatment could be halved ; it was, however, still excessive. The refined gasoline even then frequently became yellow or milky after 15 minutes' exposure to sunlight. To overcome the excessive evaporation a continuous treating system was installed. The mixing chambers consisted of four 12-ft. lengths of 12-in. pipe equipped with stuffing boxes and packing glands carrying 2-in. shafts with offset mixing blades. These blades are rotated at 250 r.p.m.. The first two cylinders are used for caustic soda washing. The gasoline leaving the second cylinder passes into a settling tank and from there into the third and fourth mixers, together with the sweetening solution. Eventually the sweetened gasoline is discharged into

an agitator a few inches above the cone, where the treating solution separates and flows by gravity back to the tank from which it was circulated. Although this equipment does not materially affect the chemical consumption, evaporation losses are now reduced to 0·5 per cent. Shortly after this equipment was put in operation, experiments were made with a view to obtaining still better agitation by introducing air. The results were astonishing and, as the volume of air which could be used without undue evaporation in the agitator was limited, it was realised that the benefits observed were due to other causes than more efficient mixing alone. The introduction of the air resulted in the immediate reduction of the litharge used to 10 per cent. Experience showed that 8 cub. ft of air per minute were sufficient for sweetening 220 barrels per hour. As lead sulphide (in the spent plumbite) was also being recirculated with the doctor, it was decided to examine the reaction in the laboratory in order to determine the parts played by the various substances present. These experiments showed that lead sulphide which had been washed free from hydrogen sulphide and suspended in caustic soda would sweeten gasoline provided that air was present. It was also found that the addition of sulphur was unnecessary, that no reduction in the sulphur content occurred, and that the reaction would not proceed in the presence of sodium sulphide. A new process has therefore been developed. Lead sulphide is first prepared by adding sodium hydrogen sulphide solution (from the soda washing of crude distillates) to plumbite, care being taken that the plumbite remains slightly in excess. The strength of the caustic soda carrying the sulphide varies from 10–25° Beaumé, but a solution of 20° Beaumé gives the best results. The treating is carried out as follows :—

The gasoline is first soda washed so that it is free from hydrogen sulphide, and the effluent contains only sodium sulphide and no caustic soda. This is carried out in the first two cylinders described above. After settling, the gasoline, together with about 50 per cent. of the lead sulphide suspension in caustic soda, passes into the third mixer. Eight cubic feet, of air per minute are also introduced and the whole mixture passes through the third mixer into the fourth, and is then discharged into the agitator. The lead sulphide suspension passes back into the storage tank for recirculation. It was found possible, passing 125 barrels gasoline per hour through the mixers, to maintain the 50 per cent. gasoline suspension mixture, using only 25 barrels of the suspension. If it is necessary to add litharge (*i.e.*, when the caustic soda shows a trace of sulphide) or sulphur they are added to the suspension in the storage tank. The latter is added whenever litharge is added to convert any mercaptides into lead sulphide. Approximately four barrels of soda are added per year to the lead sulphide suspension. New treating mixture is prepared two or three times a year. When the agitator is full it is allowed to settle 30 minutes and then dropped.

The action of the lead sulphide is undoubtedly catalytic, although it has been found that a small amount is oxidised to plumbite. The oxidation is due entirely to the oxygen of the air, which oxidises the mercaptans (probably when adsorbed on the lead sulphide) to disulphides, thus bringing about "sweetening." Although sulphur assists in this process, it is unnecessary.

S. F. B.

18. PROCESS FOR THE HOT DESULPHURISATION OF GASES DERIVED FROM THE DISTILLATION OR THE PYROGENATION OF SOLID, LIQUID OR OTHER COMBUSTIBLES. *Soc. Ind. des Procédés Prudhomme Houdry.* E.P. 267,138.

Catalysis is practically inoperative without previous desulphurisation of the gases before being submitted to the action of the catalyst in the forma-

tion of liquid synthetic fuels. The problem of the elimination of sulphur is complex, as it is necessary not only to eliminate the mineral sulphur, but also the organic sulphur. The process consists of maintaining the gases at a temperature of 250° C. in the presence of a metal or metallic oxide such as nickel oxide, in order to remove the major portion of the hydrogen sulphide. The organic sulphur is then removed by passing the gases over an oxide capable of decomposing disulphides, mercaptans and thiophenes. In practice the purifiers are employed in two parallel groups in series, thus permitting the continuous operation of the process. A plant is described capable of purifying gases containing or not containing steam, and of regenerating the catalyst. R. P.

See also Abstracts Nos. 19, 59 and 60.

Natural Gas.

19. Natural Gasoline as Chemical Material. Kaye. *Oil and Gas*, 1927, **26**, 29.

After the recovery of the higher boiling constituents of casinghead gasoline, the residue of methane and ethane which is generally burned may be chlorinated to give methyl chloride, methylene dichloride, chloroform and carbon tetrachloride. The most illustrative method of so doing consists in passing the gas and chlorine mixture preheated to between 200-400° C. over coke or activated carbon which has been treated with zinc chloride. The products above mentioned are obtained in practically theoretical yield along with hydrochloric acid. Since the reaction is exothermic, provision must be made for cooling the reaction chambers.

The methyl chloride formed can be hydrolysed to produce the corresponding alcohol.

A process of "constructive conversion" consists in preheating the above residual gases to between 400-950° C., and passing them under pressure over catalytic agents such as zinc chloride, activated carbon, elementary metals or mixtures of metallic oxides. The products are condensed and the uncondensed portions scrubbed with sulphuric acid, from which the alkyl esters are subsequently hydrolysed by dilution with water and the alcohols distilled off. Ethyl, secondary butyl, isopropyl and secondary amyl alcohols are thus produced.

The liquid constituents are high in olefin content and their potentialities as sources of glycerols, aldehydes, ketones and acids are being investigated.

The actual natural gasoline, as opposed to the residual gases, contains propane, butanes, pentanes, hexanes, heptanes and higher hydrocarbons. This may be fractionated into a propane-butane mixture, which may be compressed in cylinders for use in stoves. It burns with a smokeless, odourless, flame of cal. value equal to 100,000 B.Th.U. per liquid gallon. This gas may also be used as a substitute for acetylene in welding, and as a refrigerating material in the heat absorbtion type of refrigerator.

The butane fractions may be used for enriching water gas. If evaporated in an enclosed heat exchange unit by the gas which it is afterwards to enrich, the amount necessary for the enrichment will reduce the dew point of the mass, both by cooling and by the increase in volume when vapour is added to gas, that liquid water can be removed.

The remainder of the gasoline has an I.B.P. of 130° F. and F.B.P. 350° F., and may be used as a rubber solvent or aviation fuel. A. R. S.

20. Chief Factors Determining the Efficiency and Safety of Vapour Recovery Systems. F. V. Long. *Oil Age*. **24**, 12.

A vapour recovery system will consist of the tank and pipe lines, various controlling mechanisms, a compressor plant and an absorption unit. The

last mentioned are standard equipment and need no description. The controlling valves, however, provide a more serious problem owing to the extremely small pressure changes which occur. As the safety of a tank depends entirely on the use of the correct vacuum and pressure valve, safety valve and flame arresters, careful consideration should be exercised before making a specification.

The degree of internal and external pressure which a tank will stand may be determined by a knowledge of the thickness of the sheet and reference to tables. The next point to be settled is the maximum gas flow which the vacuum and pressure valve will be required to give. With these two conditions settled, it should be possible to choose a suitable valve. Only one such valve must be fitted on each tank, as if two or more are used there is a danger of the vacuum pallet of one and the pressure pallet of the other funtioning at the same time thus giving a gas circulation through the tank. Arrangement is made so that when the vacuum pallet opens, dry gas (hydrocarbons) and not air is admitted to the tank.

The regulator which actuates this valve must be extremely sensitive owing to the small differences of pressure which the safety of the tank makes possible. The regulator will usually be required to operate at about half to one inch of water pressure.

Furthermore, it will be necessary to fit safety valves made to function just outside the pressure limits of the vacuum-pressure valve in case the latter should stick or should not be able to handle of sufficient volume of gas. This last case may occur for instance if the gas trap on a crude line fails to function and large volumes of gas are pumped into the tank. The pressure-vacuum valve is designed only to accommodate the breathing of the tank, and would be quite inadequate in a case like this.

For protection from fire, flame-arrestors are placed after both valves. An ordinary gauze screen is not satisfactory, and, moreover, tends to decrease the flow capacity of the valve. Care should be taken that the arrestor will permit of an adequate flow. P. D.

21. Propane and Butane to Enrich Gas. Odell. *Oil and Gas J*, 1927. **26**, 31.

A water gas assembly using West Virginia coal as fuel was normally run to produce water gas, but no carburation of gas oil was made. Instead, the gas was enriched with natural gasoline (propane and butane) to an amount of 9·2 per cent. by gas volume to produce 565 B.Th.U. gas. The gasoline was drawn under its own pressure of 35 lbs. and vaporised in a metal tube heat exchanger.

The constants of the gas were as follow :—

	Finished gas.	Lean Gas. Blue gas + blow run gas.
Co_2	7·72 ..	8·6
Illuminants	·88 ..	1·4
O_2	·40 ..	·0
Co.	22·62 ..	26·4
H_2	29·00 ..	32·7
CH_4	6·3 ..	5·1
Butane (enricher gas) ..	9·18 ..	—
N_2	23·90 ..	25·0
B.Th.U./cu. ft.	565	

In the use of this gas (1) no complaints of service or gas quality were made, (2) the quality of the gas could be maintained, (3) there was no condensation of enricher as shown by the fact that the cal. value at the outlying parts of the system was slightly higher than at the works (due probably to condensation of naphthalene), (4) The luminosity of the flame was only slightly less than that of the normally carburetted gas, (5) the gravity of the gas was slightly higher, (6) the amount of enricher gas per 1,000 ft. of lean gas is less than the amount of gas oil gas required to bring the finished gas to the required cal. value, (7) the small amount of tar formed is of high specific gravity, and, therefore, settles rapidly.

The items tending to increase the cost of producing this gas are the greater amount of fuel required, the absence of the greater part of the tar otherwise formed in using gas oil and the cost of storing the gas enricher gasoline under pressure. A. R. S.

22. Improved Washing Apparatus Involving the Intermixing of Gases and Liquid. E. L. Pease. E.P. 278,105. October 6, 1927.

The invention relates to apparatus for washing gas with liquid by intermixing the two fluids, in which each of the vertical washing or mixing pipes is arranged to be in communication with the interior of two superposed lower and upper drums or receptacles, and is traversed by a moving member that carries the plates or discs. The movable disc supporting member and the associated discs are arranged after the manner of an ordinary chain pump that embodies rise and fall pipes, one of which pipes is of such a bore that the discs as they pass through it raise the liquid from the lower to the upper receptacle, while the other pipe is of such a bore that allows gas delivered at its upper part to descend with the liquid into the lower receptacle. A modification is effected by admitting the gas into the lower receptacle and allowing it to flow counter-current to the liquid. The mixing pipe may be provided with projections on its inner surface, whereby the discs are caused to tilt in their passage down the mixing pipe. If the pump pipe delivers liquid to the upper receptacle at too great a rate to be dealt with by one mixing pipe, another may be installed containing a hanging series of discs on a stationary central rod or chain. S. W. C.

See also Abstracts Nos. 30, 64, 67, 68, 131, 132, 133, 135 and 137.

Motor Spirit.

23. Stellarene: A New Motor Fuel. *Pet. Times*, 469, 10.

Stellarene is a cracked fuel or gas oil produced in the vapour phase, in the presence of a catalyst, the nature of which has apparently not been made known. It is stated that it is an anti-knock fuel, the equal in every respect to ethyl gasoline, and in some respects superior to it. Many cars in the United States could be adjusted to give a much better performance, but then the only available fuel of suitable anti-knock value would be ethyl gasoline. Now, as the supply of benzol blends and anti-knock fuels would not meet the demand, stellarene would provide another source of supply, besides giving more freedom to the trade if produced in large quantities. Its odour has been stated to be unsatisfactory, owing to the presence of mercaptans, but these can be completely removed by caustic soda and hypochlorite solution. No complaint has been made regarding its gumming tendencies. The original stellarene plant is producing 200 barrels per day, and the product is mixed to the extent of 20 per cent. with straight run spirit, and sold as an anti-knock spirit. Two other units of larger capacity have recently been commissioned. R. P.

24. Effect of Sulphur in Gasoline on Wrist Pin Corrosion in Automobiles. Diggs. *Ind. Eng. Chem.*, 20, **1**, 17.

A Jordan engine was run for successive periods on gasoline fuels of 0·151 per cent., 0·040 and 0·458 per cent. sulphur, respectively. It was found that wrist pin corrosion did not occur when the temperature of the surrounding atmosphere was well above 32° F., but that below this temperature corrosion became serious, increasing with the sulphur content of the fuel, but at a higher rate.

As sulphuric but no other corrosive acid was found in aqueous solution in the crank case along with ferrous sulphate and sulphide it is presumed that the corrosion was due to this cause.

An explanation of the fact that little or no corrosion occurs with fuel of less than 0·1 per cent sulphur is given, by presuming that the acid formed is used in neutralisation of the finely-divided iron and road dust always present in the crank-case.

The authors think that 0·1 per cent. sulphur in gasoline is the maximum allowable. A. R. S.

25. Sulphur in Gasoline from the Automobile Standpoint. Mougry. *Ind. Eng. Chem.*, 20, **1**, 19.

Almost all the corrosion and wear in automobile engines is attributable to sulphuric acid solution in the crank-case oil. Figures are given to show that dilution of the oil, even up to 50 per cent. with kerosine, will not cause undue wear provided the pressure is below 286 lb. / sq. in.

Fuel of 0·10 per cent. sulphur will cause corrosion in winter time if the engine is used intermittently. If thermostats be fitted along with ventilators on the crankcase, fuel of 0·25 per cent. sulphur can be used without detriment, especially if oil filters are in use. Any arrangement to prevent moisture condensing in the crankcase will tend to prevent corrosion. A. R. S.

26. Method of Refining Cracked Naphtha. R. A. Halloran. *Oil and Gas J.*, 1927, **26**, 29.

The method of treating cracked naphtha with concentrated sulphuric acid at low temperatures causes a considerable saving in the quantity of low-boiling product obtained, as polymerisation is inhibited to a large extent.

The crude naptha is first washed with soda and then dried with the spent acid returned from the system. This is necessary as in the following cooling any water present will separate as ice. The settled naphtha is now cooled to —5° F., and then mixed with acid in one or more stages by means of centrifugal pumps. The distribution of the acid in the washers is regulated to give the maximum desired temperature at the particular stage, and cooling is resorted to between stages. The naphtha is then water and soda washed and re-run with fire and steam. A. R. S.

27. Sulphur Content of Motor Fuels. A. S. Kraemer, E. C. Lane, and C. S. Luce. *Oil and Gas J.*, 1928, 26, **33**, 78.

The authors examined 130 samples of motor fuel now on the market. Of these only 7 had a sulphur content greater than 0·10 per cent; 5 samples had exactly 0·10 per cent. The gravity shows that half of these 12 samples are benzol blends. Twenty-seven samples were positive to the doctor test, and of these only 3 were among the 12 high-sulphur samples. In the corrosion test, only two samples are reported as showing discolouration, and both these are among the high sulphur samples. The most important conclusion to be drawn from the results is that the sulphur content of more than 90 per

cent. of the gasoline represented by these samples is less than 0·10 per cent. It further confirms that the doctor test is of no importance as a criterion of either sulphur content or tendency to corrode copper. S. W. C.

28. Modern Motor Fuels. W. A. Whatmough. *Automobile Engineer*, 1927, **17**, 236.

The results given under the same title the previous month are summarised as follows :—

	Anti-knock value	Mean volatility.	Ease of starting.
Red Line Super	Excellent	Excellent	Excellent.
Shell	Good	Excellent	Excellent.
Power	Good	Fair.	Poor.
Pratt's Perfection	Fairly good	Excellent	Excellent.
Glico	Fair.	Good	Good.
R O P	Fair	Poor	Bad.
B.P.	Fair	Excellent	Excellent.
Blue Bird	Poor	Good.	Good.
Dominion	Poor.	Good.	Good.
Pratt's Ethylised Petrol	Excellent	Good	Good.
Red Line Benzol Mixture	Excellent	Fairly good	Good.
Pratt's Benzol Mixture	Excellent	Excellent	Excellent.
National Benzol Mixture	Excellent	Excellent	Excellent.
Red Line Commercial	Excellent	Fair	Fairly good.
Pratt's Gasoline	Good	Good	Good.
Mex	Good	Fair	Good.
Blue Bird Commercial	Fair	Fair	Poor.
B.P. No. 3	Fair.	Good	Good.

W. N. H.

29. Process of Desulphurising and Purifying Oil. R. Cross. U.S.P. 1,654,581. Jan. 3, 1928.

The process is particularly intended for cracked distillates containing objectionable amounts of sulphur. A tower is described in which a solution of metallic salts flows counter current to an ascending stream of oil. The treating solution consists of certain salts of lead, copper, antimony, zinc, manganese, etc., which have the ability of combining with the sulphur present in the oil. These salts may be dissolved in water, alcohols, glycerine, ethylene glycol, etc., solvents which do not readily dissolve in the oil. The most satisfactory solutions are sodium plumbite, and copper sulphate, preferably in a neutral or weakly acid solution. The oil, if necessary, may be finished by passage through a tower filled with bentonite, fullers earth, etc., or the oil may be subsequently treated with acid and soda. The process is worked under certain temperatures and pressures, and may also be used in cracking still towers for vapours under pressure, or while the oil is in the vapour state.

R. P.

30. Method and Apparatus for Making Gasoline. F. E. Gilmore. U.S.P. 1,649,345. Aug. 5, 1924.

An invention is described, relating to the manufacture of gasoline and particularly to the condensation and stabilisation of gasoline produced in an absorption plant.

Absorption oil is treated in a still, and the products of distillation passed into the base of a tower containing baffling elements through which the

vapours ascend and over which the condensate flows in contact with ascending vapours to collect in the base of the tower.

Connected to the top of the tower is an accumulator tank, a compressor and a condenser being interposed between. Condensate from the accumulator tank flows through a pipe and is introduced into the top of the tower through spray heads, trickling down over the baffles and absorbing gasoline from the rising vapours as it does so. The pressure in the tower is much less than in the accumulator tank and is regulated by means of a back pressure valve interposed between the compressor and the tower.

Connected to the accumulator tank is a valve controlling the residue pipe regulated by a thermostat, so that when the temperature within the tower falls too low the pressure on the condensate in the accumulator tank is reduced by the opening of the valve to release some of the residue gas. Decrease in the amount of condensate sprayed into the top of the tank causes the temperature to increase to a point where the thermostat automatically lessens the escape of residue gas, thus increasing the amount of condensation and the amount of condensate sprayed into the top of the tower.

Automatic regulation of temperature is thus maintained at a substantially constant point. The result of all the controlling factors is a stable product collecting in the bottom of the tower, which above a certain depth pass from the tower through a look-box end pipe to suitable storage tanks. H. S. G.

31. Production of Hydrocarbons from Oxides of Carbon and Hydrogen. J. Y. Johnson, communicated by I. G. Farbenindustrie Aktiengesellschaft. E.P. 279,347. Nov. 27, 1927.

The formation of hydrocarbons by this means in presence of a catalyst leads to an undesirable evolution of heat. Such an evolution is controlled by mounting the catalyst in a heat exchange, whereby high pressure steam is produced. W. N. H.

32. Sulphuric Acid Recovery. C. Still. E.P. 277,619. May 16, 1927. Conv., Sept. 20, 1926.

The process is for recovering sulphuric acid from the acid-tar of benzol purification, in which the acid-tar is mixed with concentrated aqueous ammonium sulphate solution, and heated until the previously dissolved acid resins are completely separated out as a pitch-like mass. At the same time any hydrocarbons are driven off as vapour and condensed. The heating is then stopped and when cool the hardened acid resin is skimmed off and the ammonium sulphate liquor containing the recovered sulphuric acid returned into an ammonia saturator. H. S. G.

33. Sulphuric Acid Recovery. C. Still. E.P. 281,547. May 31, 1927. Conv., Dec. 22, 1926.

The patent describes an improvement of the process of recovering sulphuric acid from the acid-tar of benzol purification according to E.P. 277,619 characterised in that the acid-tar is agitated with a warm aqueous concentrated solution of ammonium sulphate without the application of external heat. The mixture after suitable agitation is allowed to rest until the acid resinous products are separated out as a layer floating on the surface of the liquid. The ammonium sulphate liquor is removed and the resin remaining in the vessel is heated until the benzol hydrocarbons dissolved therein have been driven off. Tar, tar-oil or solvents may be added to the resinous mass in the vessel before, during or after the heating, and after the resin-tar mixture has been freed from benzol it is washed with water before being run off.

The principal parts of an apparatus suitable for carrying out the above are as follows: Two mixing vessels for regenerating the sulphuric acid surrounded by an iron frame and a platform; an alkali lye washer; a cooler for cooling the gases and condensing the vapours withdrawn from the mixing vessels and a box for receiving and separating the sulphuric acid recovered.

The mixing and regenerating vessels are closed by covers provided with manholes and inlet pipes. In the central axis of each cover is mounted a vertical shaft carrying at its lower end an agitator. The shaft is driven by a bevel wheel gear operated from a horizontal shaft provided with a hand crank. In the lower part of each mixing vessel is a worm for heating the contents of the vessel without contact with steam. On the side of each vessel near the cover is a common supply pipe and a common discharge pipe for the gases and vapours. Discharge outlets are provided at the bottom of the mixing vessels for recovered sulphuric acid and resin respectively. The vapours from the mixing vessels enter a washer from which they are discharged to a condenser and led into a container for separating the water and benzol.

H. S. G.

See also Abstracts Nos. 17, 56, 58, 63, 95, 102 and 119.

Kerosine.

34. THE USE OF KEROSINE AS A REDUCING AGENT. *Pet. Times.* 19, 471, 118.

Considerable work has been carried out dealing with a process for the reduction of metallic oxides based upon the supposition that a paraffin oil at the moment of cracking would be extremely active as a reducing agent. A revolving cylinder containing the metallic oxide is heated to a suitable temperature, which in the case of zinc is between 1000° C and 1200° C. Inside the cylinder is a water-cooled tube through which the oil is allowed to flow into the cylinder. The cracking of the kerosine gives nascent carbon, and possibly other nascent elements or compounds, which quickly effect reduction. From the results of experiments it would appear that for each metal there is an optimum temperature most suitable for reduction. When a mixture of oxides is being dealt with, it usually happens that the whole of one metal is reduced before any of the remaining metals are affected. The process has been applied to numerous metals, but only in the cases of iron and zinc have the operations been conducted on a commercial scale.

W. H. T.

35. IMPROVEMENTS IN THE PURIFICATION OF HYDROCARBONS AND THE LIKE BY LIQUID SULPHUR DIOXIDE. Allgemeine Gesellschaft für Chemische Industrie. E.P. 268, 726. Nov. 17, 1927.

The invention consists in a process for purifying hydrocarbons with liquid sulphur dioxide according to the Edeleanu process, consisting in preceding the main treatment with a portion only of the liquid sulphur dioxide which is small as compared to the main portion of liquid sulphur dioxide during the subsequent main treatment, and separating the extract. It is claimed that by this preliminary treatment the whole of the moisture is removed.

S. W. C.

See also Abstracts Nos. 11, 17 and 97.

Fuel Oil.

36. Generating Gas from Heavy Fuel Oils. *Petroleum Times*, 18, 467, p. 1192.

The article is a description of the Hakol-Zwicky process of producing gas from oil, whereby the difficulty of carbon deposition is overcome. The process works efficiently on heavy grade fuel oils, and gives a clean and stable gas which can take the place of town gas for industrial purposes.

The filtered oil is passed through a preheating coil in a hot water chamber at 160°-190° F, and then is injected with a regulated quantity of air into a horizontal combustion chamber lined with firebrick. Here the oil is subjected to partial combustion and is converted into gas and light vapours. A feature of the apparatus is the avoidance of carbon formation by the injection of the oil and air along the central axis of the chamber, and the introduction of secondary air at the opposite end of it, this serving as a cushion against which the advancing column of flame strikes and is turned back along the chamber walls without actually touching them. The combustion of part of the oil produces the heat to crack and gasify the remainder of the oil. The gaseous mixture passing out of the chamber consists of hydrogen, methane, carbon monoxide, and some unsaturated hydrocarbons as well as nitrogen and carbon dioxide. Small amounts of finely-divided carbon and tar particles are also present in the form of a tar mist, but if the gas is to be used direct from the generator, these are not detrimental. If a clean gas is required, the gas is filtered through a duplex asbestos filter to remove the carbon particles, and then passed through a Smith's tar extractor under slight pressure. The gas then receives a slight water wash which removes the small quantity of condensible hydrocarbons. W. H. T.

Lubricating Oil and Wax.

37. Some Modern Methods of Recovery of Lubricating Oils. A. J. Broughall. *J.S.C.I.*, 1927, 1096–8, 1112–7.

The impurities usually found in used oils are water and particles of metal, grit and carbon. Oils from internal combustion engines may be diluted with the heavy ends of the fuel and in some cases the impurities take the form of difficultly removable colloid suspensions. As a rule, oil does not lose its lubricating property by use and a case is quoted in which the viscosity of an oil recovered and purified eight times became slightly increased. The settling tanks made by Industrial Waste Eliminators, Ltd., are provided with steam coils and reclaimed oil, water and residue draw-off arrangements. A heavy oil is maintained at 170–180° F. for two weeks, whilst the period of settling in the case of lighter oils is one week at 150° F. Soda ash is added to an acidic oil and brine to an oil containing fine particles of grit and carbon.

The Sharples Super-centrifuge is designed to run at high speeds, 15,000 to 17,000 r.p.m., developing a centrifugal force approximately 17,000 times that of gravity. The dirty oil heated to 150° F. and mixed with sodium hydroxide to neutralise acidity is fed in at the bottom of the bowl. Three layers are formed, pure oil being in the centre, solid matter in the outer, and water in the intermediate layers.

The Alfa-Laval centrifuge comprises a cylindrical bowl, which has a conical top, is of a special floating, self-balanced construction, and revolves at 6,000 to 9,000 r.p.m. The oil to be reclaimed is fed in at the top of the bowl, and passes down a tube to the bottom, which is water-sealed. The oil is at once distributed between a number of discs in fine films through a series of holes,

the solid matter being deposited on the periphery of the bowl, the water and oil forming the intermediate and inner layers respectively and being discharged through separate pipes. In the case of diluted oils, a special plant to remove the diluent and to restore the original viscosity is provided.

A means of purification is also to hand in the Stream-Line Filter. The basic feature of this apparatus is that the liquid is brought against the edges of a pile of impervious papers, the solids being arrested at the edge and the clear liquid passing between the leaves. The degree of fineness of the filtering medium can thus be varied by regulating the pressure on the pile or pack of papers. Colloidal carbon may be eliminated from used lubricating oils by this means.

A number of analyses are given showing the results of tests on recovered oils. H. G. S.

38. Wax and its Manufacture. G. W. Cupit, junr. *The Refiner*, 1927, 6, 12, 88–94.

It is impossible to make the best fully refined wax from a poor paraffin distillate. There are various crudes which give better wax than others, and should be used when the production of good waxes is desired. Paraffin distillates may be obtained by following two general methods of distillation: first, by running directly from the crude oil; second, by topping the crude, treating the reduced crude with 66° Bé sulphuric acid, neutralising, washing, and then redistilling to get the paraffin distillate stock. The oil chosen for this work was Eureka Pennsylvania Crude, having a gravity of 42·5 Bé and possessing a pure paraffin base. Paraffin distillates made from Pennsylvanian crudes are of such light gravity that a washing previous to chilling and pressing is unnecessary.

An important point to keep in mind when first chilling paraffin distillate is not to chill too fast before the formation of crystalline wax takes place. A small percentage of low melting-point wax added to the paraffin distillate speeded up the formation of crystals, but on pressing the crystals were not of the best quality. The activator which finally gave the best results was foots oil. This caused the formation of small, pure crystals, and in a much shorter time than the low melting-point wax. The paraffin distillate is first pumped through a cooler filled with river water. This lowers the temperature of the distillate from 95–100° F. to 88–90° F. with river water at approximately 82° F. A second cooler, connected to the first is used and brings the temperature down to 84–86° F. The distillate now passes through preliminary Carbondale chilling machines, where it meets pressed oil at 40–45° F. flowing in counter-current, and leaves there at a temperature of 70–72° F. The machines may be used in series. The pressed oil is pumped through the machine from an underground tank which acts as a receiving tank for the presses, and on leaving the machines it goes to storage. After passing through the preliminary chillers the distillate goes into a small storage tank from which it is continually pumped through the cold brine-chilled carbondale machines. These machines are connected in parallel with a two-inch connection from a four-inch header and a ¾-inch bypass, by means of which the feed to each machine is regulated. The pressure on the main is 300–400 lb. The distillate leaving the machines is kept at 12–14° F., and this gives a cold test of 21–22° F. The temperature of brine ($CaCl_2$ 28–20° Bé) entering these machines must be kept within one or two degrees of zero and the leaving temperature six degrees higher. The brine-chilled distillate flows into a second reserve tank, and from there is continually pumped to the presses.

After the press has been dumped the head is pushed forward by means of a hydraulic ram, the final pressure being between 950 and 1,000 lb., to insure

a tight gasket joint between rings and plates. The apron on each side of the press is then cleaned of all wax, and a trough laid directly beneath the centre of the press to catch the pressed oil which flows into underground tanks. Next the valve from the filling pump is opened into the empty press and the press is kept on this pump at a pressure of 150 lb. for an hour or so and then the valve is closed. The valve from the pressure pump is opened and the pressure increased to 300–350 lb. and kept constant for 12–20 hours or until a hard cake of wax is formed. The flow of pressed oil and the cold test are reliable guides as to when the press is ready to dump. If run too long the flow of oil will decrease to less than 5 barrels per hour. When ready to dump, the pressure valve on the manifold is closed and the pressed oil trough is pushed to one side. The screw is turned back in order to carry any oil collected there into the cellar. When clear it is reversed, so that the wax cakes, as dumped, will be carried to the dump tanks. The head of the press is pulled back the length of the ram and the plates separated from each other by placing a paddle between ring and plate and using it as a lever. The wax obtained from the press is known as "slack wax."

If the "slack wax" is dirty it is sent to the treating plant and washed with hot milk water until clean, after which it is ready for sweating. The spouts of the pans are raised and the pans are filled with water to the level of the ¼-inch galvanised wire mesh screen, which is approximately an inch above the bottom of the pan. The pans are then filled to the top with slack wax. It is then chilled down to a temperature of at least as low as the melting-point of the lowest cut made. This is done partly atmospherically and partly by cold water circulating through coils in the middle of the wax cake. Chilling must not be allowed to proceed too far, otherwise a much longer time will be used in sweating than is necessary. The temperature of the room and the water pumped through the coils is raised just fast enough to sweat out all the intermediate grades of wax without melting down the yield until the last. Toward the end of the run the oil content of the wax in the pans and the colour of the stream are noted. When the dirt and oil have been removed, steam is turned into the pans and the oven melted down. The spouts are lowered, allowing water to flow away from the pans, and this is followed by the first cut of the wax charged. As sweating continues the stream is sampled for melting-point, and when the upper limit is reached it is switched into a different drip. Before the pans are again filled they are thoroughly flushed down with water. The wax thus obtained is called "crude scale wax." This can be again charged into the ovens and run for "sweated wax." The second and first intermediate scale waxes are re-charged to the ovens and melted down for sweated wax. If the sweat oil contains crystalline wax this may be recovered by cold settling.

The sweated wax from first, second, and third intermediate scales is then given an acid treatment to remove organic matter, after which the colour obtained in filtering is permanent. As far as possible the sweated waxes are blended before treatment to make the required melting-point, but blending may be done after treating and filtering. Some refineries acid-treat the wax distillate, neutralise and wash it prior to pressing, in which case the treating of the sweated cuts is omitted. The former method gives better results and the stability of the products to both light and heat is greater. S. W. C.

39. How Water-white Medicinal Oils are Made. H. L. Kauffman. *The Refiner*. 1927, 6, 12, 70–72.

A grade of water-white medicinal oil, which when finished has a Saybolt viscosity of 210–215 seconds at 100° F., is made from Peruvian crude distillate. A narrow cut from the crude having viscosity of 200–215 seconds

at 100° F. is first made and reduced to a bottoms having a viscosity of 440–450 seconds at 100° F. These bottoms are then pumped to agitators, where the oil is treated with a total of 37 per cent. by volume of sulphuric acid, in five dumps, the first two being 8 per cent. each and the other three 7 per cent. each. The oil is cooled to 60° F. after each acid treatment—that is, after the sludge has been settled and drawn off. The oil is cooled by circulation through a chilling machine, wherein it is cooled by cold brine. The oil is then neutralised with 20° Bé caustic soda by agitation, the waste alkali being settled out. The neutralised oil is then distilled, leaving about a 4 per cent. bottom. The distillate is returned to the stills, where it is sweetened with steam at about 450–500° F. The oil is next pressed at 16–18° F. below zero to remove the paraffin wax, giving an oil of cold test from +8 to +10° F. The pressed oil is then treated with 15 per cent. of sulphuric acid in two dumps of 8 and 7 per cent., being cooled to 60° F. before the application of the last dump. The oil is then air-blown to remove the acid fumes, and transferred to stills where it is reduced to the proper viscosity and steamed sweet. Finally the oil is filtered through a column of coarse-mesh fuller's earth, giving a finished water-white product.

Another refiner mixes 50 per cent. of each of 37° pressed distillate and ice machine oil, which is then reduced to a bottoms of 120 seconds at 100° F. This is then treated in several dumps with a total of 20 per cent. by volume of fuming sulphuric acid. The oil is then just neutralised with caustic soda and, without washing, the oil is distilled down to a 5–7 per cent. bottoms. The distillate is then steamed for two or three days at 400° F. until sweet. It is again treated in several dumps with a total of 20 per cent. by volume of fuming sulphuric acid, and the excess acid removed by air-blowing, after which it is filtered through fuller's earth to give a finished product.

A patent issued to E. B. Cobb on August 16, 1921, describes a similar process, but an essential step in his procedure is the treatment of the oil with iso-propyl alcohol. This is carried-out after the neutralisation of the oil following the second acid treatment. About 5 per cent. by volume of a 70 per cent. solution of alcohol is used, and this serves to remove sulphonated compounds which otherwise seriously interfere with the filtration of the oil. A previous patent by the same man had described a process where glycerine was substituted for the iso-propyl alcohol. S. W. C.

40. Clay Applied as Pulp for Treating Lubricate Stock. G. Reid *The Refiner*, 1928, 1, 63–65.

The Texas Pacific Coal and Oil Company deals at its Fort Worth refinery with Ranger (Texas) paraffin base crude, the supply of which is controlled by the company and is free from contamination with production from other fields. This factor, coupled with exact control of oil charged to stills, regulation of speeds of all pumps, governance of temperatures of oil in stills and vapours in bubble towers, as well as steam rate to stills, insures the production of uniform products. The skimming plant equipment consists of seven 12 by 13-ft. shell stills, each equipped with Badger bubble towers, the first four stills being operated continuously and yielding gasoline, naphtha, kerosine and gas oil. The residue from the continuous battery, termed long residium, is processed in one operation to give wax and lubricating oil. It is first treated with acid (no details given) and is then ready for contact clay treatment, the clay being applied in the wet or "pulp" form, prepared as follows: The raw clay of low moisture content is pulverised so that at least 85 per cent. will pass a 200-mesh screen, and is then conveyed to a lead-lined "cooker" of 600 barrels capacity. Here it is mixed with 50 per cent. by weight of sulphuric acid and "cooked" for several hours, steam being blown through coils in the vessel. The mixture is next transferred to a cone-

bottomed tank built of California redwood, and washed repeatedly with water, the "pulp" finally obtained being a thin, pink mud. This is mixed with the acid-treated long residium in predetermined proportions in a tank from which the mixture passes to a flash or separator tank. At this point it is pumped at high velocity (1,350 gallons per minute) through a Foster tube still back to the separator, circulation being repeated until the whole is heated to just below the flash-point of the oil—*i.e.*, 400° F. (204° C.). The steam evolved escapes through a vapour line in the separator whilst the dry oil and clay are removed, cooled to 150° F., and diluted with 65 to 70 per cent. of naphtha. Maintaining the temperature, the blend is filter pressed, the pressed blend being re-heated to 150° F. with further addition of naphtha to replace loss. The next operation is dewaxing, which is effected by chilling in three insulated tanks, each of 1,000 barrel capacity and fitted internally with 200 ft. of 2-inch. pipe coils, followed by centrifuging in 20 Sharples super-centrifuges. The dewaxed blend is topped to recover naphtha, and then reduced to various specifications. The petrolatum or wax is blended with gas oil and charged to Cross cracking units. Cold exchange is also practised. H. G. S.

41. Improvements Relating to the Treatment of Lubricating Oils. Alfa-Laval Co. E.P. 279,636, 1926.

Lubricating oil which has become sludged or oxidised is mixed with the required amount of water to remove acidity and subjected to agitation in a circulating tank. The whole is then passed through one or more centrifuges, the clarified oil being subsequently filtered, if this be found necessary, and the oil returned to service. H. G. S.

42. Deodorising and Filtering Oils. V. C. Benjamin. U.S.P. 1,655,175. January 3, 1928.

In the treatment of lubricating stocks with absorbent clays it is often necessary to maintain a fairly high temperature, which may lead to decomposition, causing the oils to have an undesirable odour. The object of the present invention is to provide a simple procedure for removing such odour. Oil is placed in a treatment tank with the proper amount of finely divided absorbent clay, and is brought to a suitable treating and finishing temperature by suitable heating means. During this treatment vapours pass off through a vapour line to a condenser, the oils being kept agitated with live dry steam, which also serves to carry away the vapours of volatile hydrocarbons and all odorous decomposition products formed during the heat treatment. When the heat treatment is complete the oil is passed through pipes to the filter, steam still being passed through the oil. The filter must be of a type that will pass steam through it along with the oil, so that the steam remains in intimate contact with the oil until entirely clear of all clay and other filterable impurities. The oil and steam together pass from the filter to any suitable separator, where the steam is given ample time to separate and passes to a condenser. Should moisture show in the oil after cooling air-blowing is resorted to. H. S. G.

43. Arrangement for Refining Heavy Mineral Oils. L. Edeleanu, K. Pfeiffer, K. Gross, and P. Jodeck. U.S.P. 1,657,328. November 29, 1927.

The invention provides for an apparatus for continuously refining heavy mineral oils by means of liquefied sulphurous acid, which comprises in combination a mixing chamber provided with an agitator, a settling chamber below the mixing chamber, and a conduit leading from the bottom of the

mixing chamber to approximately the middle of the settling chamber. This conduit has a gradually enlarging discharge orifice for reducing the velocity of the flow of the fluid from the mixing chamber into the settling chamber. S. W. C.

44. Process of Treating Hydrocarbon Oils. G. Egloff and R. T. Pollock, Assrs. to Universal Oil Products Co. U.S.P. 1,649,104. November 15, 1927.

The invention consists of a process of distilling relatively heavy asphaltic and emulsified oils, of a gravity not lighter than 20° Bé, by applying heat to the oil at its upper surface by means of an electrically heated element extending above the body of the oil, and throughout the still and during distillation subjecting the oil to a vacuum in excess of 10 inches. S. W. C.

45. Process of Refining Mineral Oils. T. Hellthaler, Assrs. to Hugo Stinnes. Riebeck Montan und Olwerke Akt.-Ges. U.S.P. 1,645,530. October 18, 1927.

The invention relates to a process of refining mineral oils consisting of treating the oils with tin tetrahalogenids precipitated upon solid, finely distributed carriers (fuller's earth, kieselguhr, etc.) or distributed in an inert diluent (petroleum ether, benzene, etc.), and thereafter with a basic bleaching earth to neutralise the hydrochloric or other acid formed, this also helping to further clarify the oils. It is claimed that this process has many advantages over the sulphuric acid process and is capable of producing an asphalt-free product. S. W. C.

46. Producing Clear Water Soluble Oils. A. Horn. E.P. 281,476. July 19, 1927.

A lubricating oil capable of being mixed in any proportion with mineral oil and soluble in water free from lime in any proportion without turbidity is prepared by first heating a vegetable oil such as castor-oil to about 300° C., leaving it to cool, and then after heating it to 100–110° C. treating it with gaseous hydrochloric acid in the presence of 0·1–0·15 per cent. of a contact substance such as calcined aluminium chloride. After cooling, the oil is blown in an open vessel with oxygen, air or ozone whilst being heated from 150°–200° C., until the hydrochloric acid vapours have disappeared, whereupon after addition of a fresh contact-substance like carbonates, percarbonates or perborates of the alkalis or stannous chloride, or organic bases like resorcin, pyridine, quinone, hydro-quinone, quinoline, or hydroxylamine or trimethylamine in hydrochloric acid or alone, the treatment with oxygen, air or ozone at a temperature increased up to 200° C. is continued until the consistency of the oil has become considerably greater, the operation being terminated in four to five hours. The oil which has been cooled is then neutralised by being heated to about 50 to 60° C. with alkali or ammonia, or with an organic base, and stored for three or four weeks before use. Such an oil is particularly suitable for use as a cutting lubricant, or as a free-scouring oil for textile purposes. H. S. G.

47. Improvements in Rectifiers of Used Lubricating Oil. Rectifier Manufacturing Co. E.P. 258,838. October 12, 1927.

The invention relates to a rectifier of used lubricating oils comprising the combination of a casing having a sediment chamber at the lower portion, a division wall between the sediment chamber and the upper part of the casing, and filter means located between the inlet and discharge pipes of the casing.

The filter means is located below the removable perforated partition and consists of a cloth having a plurality of folds extending transversely in the chambers, the whole being held in place by means of a spring. The lubricant enters the lower chamber, and after passing through the filter leaves the upper chamber quite free of all sediment. It is removed from the upper chamber by means of a siphon. S. W. C.

48. Improved Manufacture of Lubricants. O. Rolfsen. E.P. 258,606. November 24, 1927.

The invention provides a lubricant which it is claimed is particularly suitable for internal-combustion engines. This lubricant substantially consists of mineral oil with an addition of fatty acid glycerides obtained from whales, seals and other marine animals. The marine oil before being added is subjected to an energetic blowing with hydrogen gas at temperatures about 100° C. in the presence of catalysts, so that partial hardening of the oil will be effected, giving it a pasty consistency at ordinary room temperatures. The proportion of partly hardened marine oil in the mineral oil can ordinarily be varied between 0·5 and 10 per cent. S. W. C.

49. Apparatus for Purifying Lubricating Oil. R. L. Skinner, Assrs. to Skinner Automative Device Co. U.S.P. 1,650,775. November 29, 1927.

The apparatus provided for is in general similar to that in patent No. 1,650,774. Here, however, the purifying and storage chambers are connected by two ports, one which opens from the bottom of the purifying chamber and is thermostatically controlled, and the other opening some distance up inside the chamber and being, at any rate partly, controlled by a float within the storage compartment. When the valve controlled by the thermostat is closed, the incoming oil is subjected to the action of the heating element, but when open, the oil escapes the action of the heating element. S. W. C.

50. Apparatus for Purifying Lubricating Oil. R. L. Skinner, Assr. to Skinner Automotive Device Co. U.S.P. 1,650,776. November 29, 1927.

The invention provides for an apparatus for rectifying impure lubricating oil consisting in the combination of a tank having an upper oil-refining chamber and a lower oil storage compartment, the two being separated by a ported wall, having valve-means controlling the port. A float is provided in the oil storage compartment, and this governs the action of the valve-means for the port, controlling the discharge of the purified oil from the chamber into the storage compartment. A heating element is placed within the purifying chamber and the impure oil after entering, encounters baffles so placed as to cause it to flow adjacent to the heating element and then into an oil-purifying space, where, by the application of suction, gases and vapours are removed. The baffles further have a thermostatically controlled by-pass, whereby under certain temperature conditions fluids traversing the chamber escape the action of the heating element. S. W. C.

51. Method of Treating Lubricating-Oil Stocks. J. W. Weir and W. J. Ryan. U.S.P. 1,650,782. November 29, 1927.

The invention relates to the removal of salts or reaction products from a sulphuric acid treated oil after neutralising with an alkaline substance, such as sodium hydroxide. If only sufficient alkali is used to just neutralise the acid remaining in the oil, then no sedimentation takes place, but the salts remain in the oil in the form of an emulsion. If now a finely divided (200-mesh) clay or other adsorbent material be added to the oil and the whole

agitated at about 300° F., the clay will adsorb the salts and after filtration the oil will be found to be free of them. Generally, clay in the proportions of 5,000 gallons of oil per ton of clay will be sufficient. It is claimed, that whilst this is not a decolourising process, that quality of the clay being destroyed by the adsorption of the salts, yet this process tends to stabilise the colour obtained by the acid treatment. S. W. C.

See also Abstracts Nos. 11, 35, 65, 82 and 97.

Special Products.

52. Dry Cleaners' Spirit. *Pet. Times.* 18, 467, p. 1,200.

The following standard specification has been prepared by the United States Bureau of Standards, and deals with Stoddard solvent used by dry cleaners. The solvent shall be a petroleum distillate conforming to the following requirements:—

(1) Appearance: clear and free from suspended matter and undissolved water. (2) Colour: waterwhite, not less than 21 Saybolt. (3) Odour: sweet. (4) Flash-point: not lower than 100° F. (5) Corrosion test: a clean copper strip not to show more than an extremely slight discolouration when immersed in the solvent for three hours at 212° F. (6) Distillation range: initial boiling-point not lower than 300° F., not less than 50 per cent. at 350° F., and end-point not higher than 410° F. (7) Acidity: the residue after distillation shall not show an acid reaction. (8) Doctor test: negative. (9) Unsaturated test: not more than 2 per cent. soluble in conc. sulphuric acid (sp. gr. 1·84). W. H. T.

53. Refining Elaterite. R. H. Haughey. U.S.P. 1,653,766. December 27, 1927.

Elaterite is an asphalt-like material used as a pigment for paint. The object of the invention is to provide a process and apparatus in which the refined material has all gas eliminated therefrom and the raw material produces its own combustion, thereby eliminating the entire expense of fuel. The apparatus comprises an open retort supported upon an air-tight container and communicating therewith by means of a restricted opening. By placing the crude elaterite in the open retort and igniting it from the top the heat of combustion causes it to liquefy and flow to the bottom of the retort and discharge through the restricted opening in the bottom into the air-tight container in which it hardens and can be removed in a few hours. H. S. G.

54. Improvements in and Relating to Pitch Compositions. Proderite Limited. E.P. 256,640. December 6, 1927.

The invention relates to the manufacture of pitch compositions which are capable of being machined, and do not break easily. The pitch employed is one having the characteristics described in E.P. 201,650 and 237,010, and should have a softening point in excess of 40° C., or other temperature related to the conditions of use. This pitch is combined with a comparatively low proportion of asbestos fibres or flakes, preferably with an addition of stone dust or similar material. The asbestos may be in the form of long or short fibres, or may be replaced by other fibres such as hemp, cellulose, or hair. The mixture usually contains from 10–15 per cent. of asbestos. W. H. T.

55. PRODUCTION OF EMULSIONS. W. A. Whatmough. E.P. 280,096. May 13, 1926.

The process for the production of an emulsion consists in mixing with a fatty oil or like liquid fatty substance a small quantity of a higher and non-volatile fatty acid, and then mixing this mixture with water containing a protein with which a small quantity of alkali has been mixed in amount requisite to make the electrolytic activity of the electrolyte or electrolytes in the aqueous phase such that the interfacial tension is sufficiently reduced to permit rapid emulsification. If a prepared protein such as casein or gelatin is used, a further quantity of alkali must be added to neutralise fermentation acids in order to bring it to the approximate iso-electric condition prior to mixing it with the small quantity of alkali. To the emulsion may be added water or a water solution, such as a neutral solution of an essential mineral salt, to thin it, or a fruit or vegetable juice and/or syrup or saccharine solution. Again, a further quantity of a fatty acid, such as oleic acid, may be added, and a wax such as paraffin wax for hardening purposes. A portion of the fatty oil or liquid fatty substance may be substituted by a hydrocarbon product, such as paraffin molle. The original emulsion, or emulsion incorporating any of the above may be evaporated under a vacuum until a clear emulsion is obtained. H. S. G.

See also Abstracts Nos. 14, 39 and 46.

Analysis and Testing.

56. DETERMINING GASOLINE VAPOUR TENSIONS. C. D. Gard. *Oil and Gas J.* 26, 29, p. 138.

In determining vapour pressures of gasoline in the standard bomb serious inaccuracies are introduced owing to the partial pressure of air enclosed in the vapour space. In the proposed method allowance is made for the entrapped air by taking one reading of the pressure and then doubling the vapour space and taking a second reading. If it is assumed that enlarging the vapour space does not alter the composition of the liquid, so that in each case the vapour pressure of the liquid is the same, we have :—

X = supposed pressure of air at first reading.
P = pressure exerted by vapour.
Total pressure = A = X + P.

At the second reading the partial pressure of the air $= \frac{X}{2}$.

Total pressure = $B = \frac{X}{2} + P$.

The $P = 2B - A$.

In order to obtain the vapour pressure of gasoline stored under pressure the sample to be introduced into the bomb was cooled by drawing through a coil placed in an ice-salt bath, and the air in the bomb was replaced by gasoline vapour before filling. Even when these precautions are taken the vapour pressure reading is low except in the case of liquids with a narrow boiling range owing to the change in composition of the liquid when a second volume of vapour is drawn off. It may be possible to obtain a correction factor to be applied in such cases. P. D.

57. THE "A" TUBE.—AN IMPROVED FORM OF "U" TUBE. Jones and Hobson. *Chem. and Ind.*, 46, 52, 1,222.

This tube is similar to the "U" tube except that the limbs are bent so that the shoulders of the long limbs are in contact. At the base are plugs inserted in the limbs which alone are filled, leaving the cross tube empty. Serious choking is thus avoided, cleaning simplified, and the whole structure strengthened. A. R. S.

58. DETERMINATION OF THE CORRODING SULPHUR IN BENZOL. Kattwinkel. *Brenn.-Chemie*, 8, 20, 259.

The corroding sulphur of motor benzol is formed in the process of thickening the tar wash oil, and in distilling the higher fractions of the pre-benzol product purified with sulphuric acid. No method is described in the literature for quantitatively determining "free-sulphur," but its affinity for metals like mercury and copper suggests a simple quantitative method of procedure. If the total sulphur content be determined, and then the sulphur remaining after treatment with a metal, the difference in results will be the percentage of free sulphur. The corroding sulphur in benzol can be removed by "Naturkupfer C," a bronze which is very finely divided and can therefore be distributed throughout the liquid. 100 gms. of benzol is heated with agitation under reflux for half-hour with 0·5 gms. of the bronze. After cooling the benzol is filtered and tested with mercury for free sulphur. Alternatively, the free sulphur may be determined by decomposing the metal sulphide formed, conducting the hydrogen sulphide into a solution of cadmium acetate, decomposing the cadmium sulphide with N/10 iodine solution, and titrating the excess iodine with thiosulphate. When mercury is used, the mercury sulphide is transferred to a stoppered bottle with some carbon disulphide, and shaken with an excess of N/10 iodine solution. The carbon disulphide dissolves the sulphur as it is formed; otherwise it would envelop particles of the sulphide and prevent its decomposition. W. H. T.

59. DETERMINATION OF NITROGEN BASES IN PETROLEUM. McKee and Parker. *Ind. Eng. Chem.*, 19, 12, 1,343.

Concentrated sulphuric acid dissolves nitrogen bases as well as unsaturateds in petroleum and shale distillates. In this paper hydrochloric and acetic acids of varying strength are tried. The method consists in agitating the acid and oil together at low temperature in a Babcock bottle and then centrifugally separating and measuring the decrease in volume. Using synthetic mixtures of amylene, hexane, pyridine, quinoline and petroleum, it was found that hydrochloric acid above 15 per cent. reacts with the unsaturated bodies present, giving results 1–2 per cent. higher than 25 per cent. acetic acid. On standing in contact with the oil overnight 10 per cent. hydrochloric acid gave the same results as conc. sulphuric acid. From the results obtained 25 per cent. acetic acid is the best reagent for use in rapid estimations of the nitrogen bases present in petroleum. It gives clearer solutions, dissolves more and more quickly the bases present, and causes less polymerization than hydrochloric acid. A. R. S.

60. A SMALL HIGH-PRESSURE AUTOCLAVE. Peters and Stanger. *Ind. Eng. Chem.*, 20, 1, 74.

The shell is of metal which is tested at 3000 lb. square inch, and into this is placed the tube which may be of glass or metal, and forms the reaction tube. This tube is filled to one-third of its total capacity and hydrogen is pumped into the shell at 5,000 lb. pressure by means of a flexible connection. The

shell is pivoted at its centre and an oscillating motion is provided by means of an eccentric pulley. The heating is accomplished by electricity and by means of a three-heat switch wattages of 1,760, 880 and 440 may be obtained.
A. R. S.

61. The Estimation of Nitrogen in Petroleum and Bitumens. Poth, Armstrong, Cogburn, and Bailey. *Ind. Eng. Chem.*, 20, 1, 83.

The use of Dumas' method in the estimation of nitrogen in crude oils may give high results owing to the distillation of the more volatile portions before combustion takes place. A modification using two connected quartz tubes and a continuous CO_2 generator, the tube nearer the azotometer being brought to red heat before the tube containing the sample is heated, is said to be satisfactory. The writers found this method tedious, and contrary to Mabery's views, found a modification of Kjeldahl's method both fast and accurate for both crudes and bitumens. 1 to 5 gms. of the sample are digested with 150 ccs. of sulphuric acid, 50 gms. of potassium sulphate, 2·5 gms. of mercuric oxide, and 2·5 gms. of copper sulphate. Thirty minutes to two hours are required to bring the colour to dark green, after which digestion is continued for five hours. Before the ammonia distillation 1 gm. of granulated zinc is added to the liquid with flask, and the soda is added through a dropping funnel, the end of which is bent upwards, while the receiver is fitted with a 1-inch mercury seal. The following data show comparison between this and the combustion method :—

	Per cent. Nitrogen by Kjeldahl.	Per cent. Nitrogen by Combustion.
Ventura Crude	·420—·429	·477
Medway Crude	·424—·432	·475
Snyder Well No. 1	·442	·513
Crane Well No. 2	·451	·403
Gunies Lease No. 4	·619	·531

Natural Bitumens.	Per cent Nitrogen by Kjeldahl.	Per Cent. Nitrogen by Combustion.
Gilsonite ordinary	2·18—2·37	2·13—2·32
Tobbyite	2·26—2·32	2·29—2·35
Trinidad Lake	·7—1·01	·78—1·12
Bermudez Lake	·88— ·91	·93— ·96
Uralde asphalte with pyrite ..	·63— ·64	·66— ·67
Asphalte from Brea, Los Angeles, Calif	1·14—2·23	1·22—2·38

A. R. S.

62. A Rotary Filter Stand. Shaw. *Ind. Eng. Chem.*, 20 1, 100.

A cast-iron base is fitted with an upright iron rod on which two concentric wooden circular stands, which are relatively fixed at a distance of 8 inches, rotate. The bottom shelf carries two concentric rows of twelve beakers each, while the second carries two sets of twelve filter funnels arranged above the beakers. A fixed top shelf carries the wash bottles. The whole apparatus can be safely used on a laboratory stool. A. R. S.

63. Determination of Volatility of Gasoline. Stevenson and Babor. *Ind. Eng. Chem.*, 19, 11, 136.

The Engler distillation gives at best only a rough idea of the volatility of gasoline. Various methods making use of vapour pressure and phase change methods have been found to be tedious and inexact. Dew point methods previously enunciated have tended to give low results, but the following

method by comparison with pure chemicals of known physical constants has been shown to be exact and simple in operation. The apparatus consists of a dropping funnel of about 50 ccs. capacity, which by means of a stopper just fits inside a copper vapourising tube 1·2 × 20 cms. in which five gauze screens are fitted at equal intervals. To the lower end of this is fitted a Dewar bulb 6·5 cms. o.d., 1·9 cms. i.d. by 20 cms. long, which carries at its lower end a glass T-piece. One end of the T-piece goes to a condenser, while the other vertical limb serves for the insertion of the black surface dew indicator. The vapouriser and to some extent the upper part of the Dewar bulb, which are both heavily lagged, are heated by a stream of hot air in a heating jacket. The dew indicator is a platinum blowpipe tip carrying iron and constantan wires soldered to it about 2 mm. from the apex. The assembly is fixed to a lead glass tube up which a blast of air serves as a cooling medium. The tip is finally coated with a deposit of platinum black. The air stream to the vapouriser is started at 230—260° C., the illuminator to the platinum black indicator turned on, and the spirit allowed to drop so that the drops are not quite separate. When the cold blast is turned on to the indicator dew is readily observed by glistening if much condensation takes place, or darkening and smoothness of the film if only a small amount is present. Having determined the approximate temperature of dew formation as above, the exact temperature is observed by cooling the tip to approximately 10° above this temperature and finishing the cooling slowly.

Air gasoline mixtures are similarly carried out using instead of the dropping funnel an air supply at constant rate while the gasoline is fed in by means of a plunger. That the first method gives equilibrium end-points corresponding to true boiling-points is shown by the table following :—

	Boiling Point °C.	Equilibrium End-point °C.
Xylol	.. 140·3	.. 140·3
Alcohol	.. 78·9	.. 79·0
Bromobenzene ..	.. 156·0	.. 156·8
Chlorobenzene ..	.. 132·0	.. 132·0

Using chlorobenzene and bromobenzene in admixture with air in partial pressures as given in the tables of Landolt and Börnstein, the following results were obtained :—

	Vapour pressure mms. Hg.	Landolt-Börnstein °C.	Dew Point ° C.
	26·68 ..	40·0 ..	38·2
Chlorobenzene ..	41·46 ..	50·0 ..	49·5
	64·74 ..	60·0 ..	60·6
Bromobenzene ..	16·92 ..	50·0 ..	50·3
	27·54 ..	60·0 ..	60·6
	43·31 ..	70·0 ..	70·0

A. R. S.

64. Hydraulic Lift for Gas Analysis Apparatus. Tanch. *Ind. Eng. Chem.*, 19, 12, 1,349.

A three-way brass stopcock connects water supply to drain and a brass tube having a piston and long rod running vertically in guides. To this rod is connected the reservoir, which by its movement in a vertical plane fills or empties the gas burette of an Orsat apparatus. Its performance is actuated by the opening and closing of the needle valves on the three-way pipe, and is claimed to be successful, particularly in combustion analysis. A. R. S.

65. The Behaviour of Oils on Solidifying and Melting. Vogel. *Erdöl und Teer.* 3, 33, p. 534.

The solidifying point of mineral oils is stated to be a very poor criterion of its behaviour on cooling, owing to the fact that part of it solidifies in an amorphous condition and part crystalline. The point at which viscosity can no longer be measured is the same as the solidifying point, but as supercooling may easily amount to 10—20° C., a much more accurate constant would be the point at which the oil begins to flow after being cooled down to the solid state. This flow point is found to be higher than the solidifying point, but the degree of course depends on the character of the oil and the paraffins present. When the temperature rises to where no crystals remain in the oil, the viscosity figure of the oil being cooled will agree with that obtained during heating. A technique for the determination of flow point has been developed by the Rhenania-Ossag Mineralölwerke. The method eliminates subjective errors, establishes the viscosity in the neighbourhood of the flow point, and measures the range of supercooling. The apparatus comprises a U-tube with one arm wider than the other, a thermometer for the U-tube, one for the heating and cooling bath, and an unsilvered Dewar vessel. The U-tube holding the oil sample is placed in an alcohol bath contained by the Dewar vessel. Solid carbon dioxide is added to the alcohol until it is cooled to −30° C. When the oil is cooled to this temperature, pressure equal to a 60 cm. column of water is applied to the oil in the wide arm. If the oil moves, cooling is continued to −40° C. and the test repeated. If no movement occurs, the pressure is maintained constant, and alcohol is added to the bath until the oil moves up the small arm of the U-tube to a predetermined point. The temperature is then read on both thermometers, and the mean of the readings taken as the flow point. Viscosity in centipoises is found by noting the time required to push the oil up the small arm over a definite distance, the tube having previously been calibrated. W. H. T.

66. Boiling-point Apparatus for Calibrating Thermocouples. T. De Vries. *Ind. Eng. Chem.*, 19, 12.

The apparatus described does away with the necessity for a radiation shield over the couple. Superheating is prevented by bubbling the vapour through a second volume of the liquid; and a precision of ·003° C. is obtainable with a base-metal couple. The apparatus is described sufficiently fully for constructional purposes. P. D.

67. Suggest New Standard for Gas Testing. Warner. *Oil and Gas J.*, 1927, 26, 28.

The use of low temperature fractionation is suggested. The removal of methane is actually not carried out by true distillation but simply by regulating the temperature so that no appreciable amount of ethane distills off. The separation of the other individual members at temperatures calculated from their vapour pressure-temperature curves. The apparatus consists of a glass burette, a series of distillation bulbs which can be cooled to the required temperatures, a pressure gauge for low pressures, and a gas transferring pump. The apparatus is evacuated before admission of the sample, from which the CO_2 and O_2 have previously been removed, in the Orsat apparatus. First of all the sample is fractionated into binary mixtures, and these are then held at such temperatures that only the lower boiling has an appreciable vapour pressure. By analysis of synthetic mixtures this method has been found accurate to within 0·1 per cent. A. R. S.

68. Apparatus for Storing and Circulating Gases. White and High. tower. *Ind. Eng. Chem.*, 20, 1, 95.

This apparatus is useful where it is not required to keep the gas in storage more than a few hours and where the gas is not affected by rubber. The storage system consists of two rubber balloons placed inside inverted glass bottles which are filled with water, the bottles being connected with each other and with a reservoir placed above on a scale pan. The balloons are connected to each other and to the pump. When the apparatus is in use gas flows from one balloon through the circulating apparatus and pump to the other balloon, while water flows from one bottle to the other in the opposite direction. Only when there is a change in gas volume does the mass on the scale pan alter. The circulating system consists of a three-way cock which feeds either balloon as required, while by means of another three-way cock the other balloon delivers gas to the inlet side of the pump. Samples may be drawn off from either reservoir. The pump has a mercury piston, and by means of a U-tube with two mercury seals the gas may only take motion in the desired direction. The apparatus was found useful in the synthesis of methane from blue gas. A. R. S.

See also Abstracts Nos. 27, 28 and 52.

Refinery Plant.

69. Using Chemicals Protects Distillation Equipment Against Corrosion. G. Egloff and J. C. Morrell. *Refiner*, 1927, vi. (12), 56.

One of the most urgent problems in the petroleum industry is the prevention of corrosion. The article considers the injection of chemicals to neutralise the corrosive substances resulting from the atmospheric or super-atmospheric distillation of petroleum oils. A number of metallurgical methods of combating corrosion have been suggested and some have gone into commercial use. Reagents which have been used for minimising corrosion are sodium hydroxide, ammonium hydroxide, sodium carbonate and hydrated lime. These are usually introduced into the system by means of direct-acting pumps, and results obtained indicate that corrosion is reduced over 50 per cent. by the use of amounts which are small in comparison with the quantities required to neutralise all the corrosive substances. Ammonia and its sulphides are unstable and decompose at the temperatures of cracking. It is used mainly to neutralise hydrogen chloride in the condenser. Sodium carbonate, while containing only 75 per cent. of the alkali content of sodium hydroxide, is much cheaper per unit of alkali. Caustic soda, soda ash, or hydrated lime is injected into the system by making up solutions of the proper concentration and regulating the amount fed. The most favourable point of introduction into a cracking system has been found to be the charging oil line which leads to the dephlegmator or the heating tubes. The solution entering the feed-line mixes with the oil to be distilled. The water evaporates from the alkaline solution in the dephlegmator and leaves the system with the oil vapours. The chemical reagent is dispersed in the oil and passes through the heating element to the vaporising chamber. As the segregation of lumps of the reagent is detrimental, a thorough dispersion throughout the oil is desirable. To accomplish this, the oil, or a part of it, and the reagent are passed through an orifice mixing-column and the mixture of oil and reagent is then passed into the raw-oil line leading to the dephlegmator. The caustic soda solution has a gravity of 10° Bé, and is injected in the proportion of 0·2 to 0·3 per cent. of the oil by volume. Some difficulty has been

observed in the deposition of the solid reagent or the reaction product upon various parts of the heated system. This is offset to a considerable extent by the use of the orifice mixer or by injection in a more advantageous part of the system. Another difficulty is that the deposition of coke in the heating tubes is increased to a considerable extent. On the other hand, corrosion is cut down 50 per cent. and one commercial plant reports that coke deposition was cut down 80 per cent. Soda ash causes less deposition of coke than caustic soda. The solution is made 10° Bé, and the proportion required is about 25 per cent. more by weight than for caustic soda. In using lime, about 0·4 to one pound of hydrated chemical lime per barrel of oil charging stock will be satisfactory. The lime may be added to a portion of the charging stock and mechanically agitated so that it remains in suspension. Some of the advantages resulting from the use of lime are :—(1) The heating tubes are more easily cleaned, as the lime apparently yields a protective covering between the steel and coke, preventing its close adherence. (2) The structure of the coke in the reaction chamber is improved so that a smaller amount of fines or breeze is produced in cleanouts and in handling. The coke is more readily broken up in the chamber. (3) The cost of operation is less than when using caustic, due to the lower cost of lime. (4) Corrosion is decreased materially. S. W. C.

70. Caustic Added to Charging Stock Counteracts Corrosion. G. Reid. *Refiner*, 1927, 6, 12, p. 54.

The Crystal Oil Refining Corporation, Louisiana, has just completed an extensive expansion and rebuilding operation at its Cedar Grove refinery. This includes the installation of a tube still and bubble towers replacing baffle equipment on the skimming plant and the shell still used for re-running pressure gasoline as well as their employment with the tube still. The charging stock is a mixture of oils from nearby fields such as Pine Island, Caddo and Bull Bayon and a large proportion of Smackover, Arkansas crude. Since much of this, especially the latter, is corrosive, due to its content of sulphur, salt, etc., the oil is charged to the preheaters with a small amount of caustic solution. The caustic used for this purpose is for the most part spent solution used at the chemical treating plant. It is taken into the pump on the suction side and discharged with the oil into the preheater tank. The amount of caustic, of 18° gravity, used is about 50 gallons to every 1,200 barrels of crude run. This is governed by frequent testing of the streams which are held slightly alkaline or showing no acid reaction. This practice has certainly retarded corrosion to a marked degree, and after six months of use the shell stills show no ill-effects due to caustic accumulation on the bottoms. Prior to this practice it was often necessary to take stills off the line about once every thirty days for the repair of condenser coils or vapour lines, due to corrosion. Present practice has virtually eliminated the frequent shut-down periods for coil repair. The charging pump discharges the crude into a preheating tank where it is preheated to about 150° F. by exhaust steam. It then passes to the heat interchanger, where its temperature is brought up to 300 to 325° F., and then into the separator where it drops any basic sediment or foreign matter such as salt, finally passing to the first still, which it enters at about 300 to 325° F. There are nine shell stills in the battery. Straight run gasoline is taken from the first five ; the sixth is cut out and used for re-running pressure gasoline ; the seventh and eighth produce kerosene ; with gas oil as the overhead product derived from the ninth or last still. Bottoms from this still are 16 to 18 gravity fuel oil and are charged to the tube still. This is a complete distillation and fractionation unit in itself, and through the use of the large bubble type tower three products

are conducted through the receiving house. These cuts are benzine, light gas oil and heavy gas oil. The residuum taken from the bottom of the tower is a marketable fuel oil, containing less than 1 per cent. free carbon. The gas oil is used for charging to the Jenkins cracking units. S. W. C.

71. Purifying and Drying Oil. The American Sheet and Tin Plate Company. E.P. 282,321, Sept. 13, 1926.

A method of purifying and drying oil separated from an emulsion of oil and water particularly adaptable to the recovery of palm oil used in the processing of tinned plates.

Untinned plates are fed through a body of flux into a tin pot and out through a body of palm oil. They are then led to a vat where they are scrubbed in an alkaline solution and pass on in normal process. Oil is removed in the vat in the form of an emulsion, and at the same time the oil is purified from flux and suspended matter by the hot alkaline solution. The vat is adapted to a constant flow of alkaline liquid which passes on as an emulsion through a heating tank into a treating tank, where sulphuric acid is added and oil and water separated by gravity. The oil is pumped out through a screen into a steam heated drying drum. The heated oil passes through a standard centrifuge and the finally purified oil is discharged into a tank or vat ready to be returned to the oil side of the tin pot ready for further use on tinned plates. H. S. G.

72. Automatically Cleaned Tar Separators. Compagnie pour la Fabrication des Compteurs et Materiel d'Usines à Gaz. E.P. 272,237. Appl. June 7, 1927. Conv. June 4, 1926.

An impact tar separator is described, characterised by a chamber constituted by alternate pairs of ring elements, assembled together by means of longitudinal bolts, so disposed as to leave between them narrow circular slits, in front of the openings of which a portion of the surface of the alternate elements constitutes impact surfaces, the edges of the slits and the surface of the impact walls being adapted to be mechanically cleaned by scrapers the shape of which closely follows that of the surfaces to be cleaned. The scrapers may be movable with respect to the fixed chamber or fixed and secured to the fixed casing of the separator, in which case the chamber is movable with respect to the fixed casing and scrapers. H. S. G.

73. Distilling Hydrocarbons. W. M. Duncan. U.S.P. 1,654, 797. Jan. 3, 1928.

This invention relates to methods of distilling liquid and solid hydrocarbons at the same time. A charge of liquid hydrocarbons is heated by a suitable fire, and heat is transmitted from the liquid to the solid hydrocarbons which may be submerged in the liquid, and a charge of the same material may be arranged in the vapour line. Both bodies are maintained at temperatures high enough to vaporize their volatile constituents, and the mixed vapours are condensed in any suitable manner.

A suitable apparatus consists of a still arranged over a heating chamber. Vapours arising from the liquid in the still pass into a vapour line which includes a pair of heat-insulated chambers, the upper ends of which are connected to a reflux condenser, the vapour line leads from the reflux condenser to a final condenser and a return pipe leads from the reflux condenser to the still. A grid is arranged in the still to support a mass of coal, and each of the chambers contains a charge of bituminous coal or other solid hydrocarbon material, supported on a grid through which the vapours are admitted. Valves suitably disposed allow the vapours to be diverted into one chamber

or the other, so that recharging can be carried out without stopping the distillation. After a fresh charge is introduced, steam is admitted to expel air. The system can be adapted for use for cracking at high temperatures and pressures.

The system produces a new motor fuel containing the volatile constituents of hydrocarbon oil and bituminous coal, this being obtained without overheating the coal from which the undesirable smoke-producing fractions are removed and converted into a valuable liquid, leaving a residue of smokeless fuel. H. G. S.

74. Treating Emulsified Oil. G. Egloff and H. P. Benner. Assrs. to Universal Oil Products Co., Illinois. U.S.P. 1,649,103. Oct. 23, 1920.

The patent describes an apparatus for treating emulsified oil, in which separation is effected by forcing the emulsion through a plurality of detachable spaced plates seated in a cylinder, by means of a reciprocating piston mounted therein. The orifices in the plates are of successively decreasing size from the inlet side of the chamber, and suitable arrangements are incorporated for drawing off the separated oil and water. H. G. S.

75. Fitting Pumps to Oil Tanks. W. L. Hart and the Anglo-American Oil Co., Ltd. E.P. 280,399. Jan. 11, 1927.

Means have been invented for fitting pumps to oil or other tanks in particular to those in which lubricating oil is stored, whereby one type of pump may be readily substituted for the other, comprising a plate with a screw threaded nut or boss and perforations so located as to register with existing perforations on the top of the tank. H. G. S.

76. Process of Dehydrating Oil. B. Hildebrand. U.S.P. 1,650,514. April 2, 1925.

A process of separating emulsified oil and water is described, which consists in compressing the emulsion and then expanding the same and causing forcible impact thereof against a heated surface, then passing the emulsion in a tortuous course through restricted spaces between particles of succeeding layers of finely-divided material, and finally settling the emulsion so as to cause all of the oil to rise through the water and in so doing being washed clean of grit and other foreign matter. H. G. S.

77. Distillation of Hydrocarbon Oils. F. A. Howard. U.S.P. 1,649,532, 1927. Appl. Aug. 12, 1920.

Heavy oils are passed in an upward direction through a heated zone to a bulk supply maintained under pressure, 3 to 7 per cent. of steam, calculated as condensed water, being injected into the oil stream at a point between the heated zone and the bulk supply. The described apparatus consists of a bank of inclined heated tubes connected at each end to headers, these in turn being placed in contact with the ends of a horizontal cylindrical still by means of pipes. An upwardly directed tip or nozzle for admission of steam is placed in the shorter of the two legs. The still is not directly heated. H. G. S.

78. Fractional Distillation Process and Apparatus. H. Koppers, Assr. to Koppers Development Corp., Pa. U.S.P. 1,646,698. July 6, 1921.

The invention consists of an apparatus for the step-wise distillation of natural or artificial mixtures having different boiling points having in combination storage receptacles, a series of vaporising devices arranged one above another, consisting of flat iron trays through which the liquid flows in a

thin layer and provided with heating means, pressure means arranged to deliver a quantity of the liquid mixture from the storage receptacles to the uppermost of the vaporising devices, and means for conducting the residue from the lowermost of the vaporising devices to the storage receptacles.

The stepwise distillation of natural or artificial liquid mixtures is accomplished by conducting the quantity to be distilled in any time in a constant current through a single distillation apparatus, in which the distillation, with the aid of reflux cooling, is divided into two sharply separated fractions. The residue of the first distilled charge, by properly regulating the temperature and the vacuum in this distillation apparatus and the reflux cooling, is again subjected to distillation and again divided into two fractions. When by suitable repetition a complete distillation of the first quantity separated has been accomplished, a new liquid quantity is subjected to the same process.

The arrangement used consists of the distillation apparatus proper and two receptacles which can be alternately connected in such a way that, during one operating stage, one of them contains the quantity to be distilled, and the other receives the resultant residue, while by setting the apparatus for the second operating stage, the task of the two receptacles is reversed.

In order to enable the residue and the two fractions obtained to flow back freely, the distillation device, the dephlegmating column with reflux cooler and the final cooler are placed at so high a point that a free flowing back into the low level storage receptacles is rendered possible at any time. H. G. S.

79. Improvements in Heat Exchange Apparatus. J. Muchka. E.P. 272,152. Oct. 6, 1927.

The invention provides for heat exchange apparatus composed of plate-shaped members which are connected together at opposite edges by means of W-shaped edge members, characterised by the feature that reinforcing parts are inserted in the W-shaped edge members for keeping the plates at the correct distance from one another. These reinforcing parts, which lie freely in the grooves, are provided on their ends with studs which engage in strips by means of which bars running in a parallel direction, and thereby the individual members are connected together, preferably in such a manner as to be capable of being tightened up. An example is shown of an air preheater working on the cross-flow principle, and built up of square plate members.

S. W. C.

80. Side-furnace Oil Still. R. A. Porterfield. U.S.P. 1,647,664. Nov. 1, 1927.

This patent covers a still of the horizontal cylindrical type, supported along its sides and at each end by a setting having a number of transverse walls, normally not in contact with the still, which will support the still bottom at intermediate points along its length if substantial sagging or deformation should occur. Separate furnaces are disposed between these transverse walls, so that by suitable control uniform heating may be applied throughout the length of the still bottom. The burned gases from these furnaces pass across the bottom of the still to a longitudinal flue disposed in the still setting.

E. R. B. S.

81. Improvements in Process and Apparatus for Evaporating Oils. C. W. Stratford. E.P. 280,727. Nov. 24, 1927.

The invention provides for a process of evaporating oil in a still, including the drawing of oil from a point below the level of the oil by a pump situated within the still and whose outlet connects up to a manifold, which in turn feeds a series of nozzles placed close to the still wall. The oil is thus discharged under pressure against the inner heated surface of the still with such force as to cause the formation of thin films of oil rapidly spreading and moving on

the heated still surface, whereby a rapid circulation of the oil is set up within the still and repeated contact of the oil with the highly heated portions of the still is produced. During charging the oil is made to flow down inclined plates, in the interior of the still, covered with netting which imparts a rippling or agitating movement to the charge, which, being spread in a thin film, becomes preheated before mixing with the body of oil in the still, thus vapourising the more volatile products from the charged oil prior to its admixture with the body of oil in the still. S. W. C.

82. Improvements in Filters. E. J. Sweetland. E.P. 275,333. Aug. 5, 1927.

This invention relates to apparatus for concentrating a thickening liquid containing small percentages of solid matter, in order to prepare the concentrated liquid for economical filtration in other filtering apparatus. The apparatus comprises a rectangular tank with a cone-shaped bottom terminating in a flanged pipe connection which forms a communication between the lowest part of the cone and a pump suitable for slowly pumping a thick sludge. Inside the tank is a plurality of headers, one end of which is connected through the tank wall to a pipe outside, and the other end closed by a cap. The outside pipe of each header carries a sightglass, and communicates with a main header running the whole length of the tank. A plurality of filter elements are connected to each header by means of nipples screwed into the header. These filter elements are preferably tubular and comprise a wooden drainage member hollow at the upper end and connected to the nipple by a casting. Each drainage member is covered by a bag of a suitable filter fabric, which is closed at the bottom and gathered at the top into a circumferential groove by means of a cord or wire. These bags are of greater size than the drainage members. For liquids which foam a foam-removing device is provided. The main header is connected to a pump of the high vacuum type, which will operate with equal facility in either direction, and discharge the filtered liquid into a filtrate tank. The pump is operated by a motor connected to control mechanism so that it can be reversed. The pump is so controlled that there are three periods per cycle of operation; first, a filtering period during which a filter cake is deposited upon the cloth; second, the pump is reversed, causing filtrate to flow back through the cloth which bulges and forces off the cake previously deposited; third, a period of quiescence during which the dislodged cake is allowed to settle to the bottom of the tank. These periods are, of course, of unequal duration.

A similar filter wherein the liquid is forced through the filter cloth by pressure is also described. S. W. C.

See also Abstracts Nos. 20, 22, 43, 44, 47, 49, 50, 53, 90 and 139, and those under the heading of "Cracking."

Engineering.

83. Steel at High Temperatures. Strength and Safety Factors. *Oil and Gas J.*, 26, 31, p. 64.

The load which any piece of plant or apparatus can carry with safety depends on the strength of the material used in the construction, coupled with the variations of resistance to strain introduced by the actual design. In applying a factor of safety to pressure containers a recourse is had to the knowledge of the behaviour of the design under stress, and the strength of the steel as determined by test.

The author believes the fracture strength of a piece of metal is easy to determine experimentally, and should be that used as the foundation for

the factor of safety. In determining the strength of steel at high temperatures it is most important to notice that the duration of the test has a great bearing on the result. Thus, experiments in which a piece of steel is submitted to a constant stress for a short time give a value of 32,500 pounds per square inch; whereas if the stress is applied for 300 hours the strength is only 15,000 pounds per square inch.

In the matter of design joints and heads of pressure vessels tend to be a weakness. It should not be forgotten that a joint which has not been thickened correctly may adversely affect the body of the vessel in its immediate neighbourhood. As the result of experiment it has been shown that in order to introduce 100 per cent. efficiency into the design of the head the shape should be one-half an ellipsoid of revolution in which the minor axis is one-half the major axis.

Corrosion is also a big factor affecting the safety of plant, and in this connection it is well to note that the fact that the metal is under stress affects the rate of corrosion. It is not wise to assume that the rate of corrosion of an unstressed specimen of metal kept under the same conditions is a measure of the rate of corrosion of the pressure vessel itself. P. D.

84. Reagents for Controlling Corrosion. Egloff and Morrell. *Oil and Gas J.*, 1927, 26, 29.

In the control of corrosion by chlorides and sulphides, caustic soda, slaked lime and ammonia have been successfully used.

The most favourable point for the injection of the reagent in cracking plants is the charging oil line leading to the heating coils or dephlegmator. To prevent the segregation of large amounts of oil in the system the reagent and part of the oil are mixed in an orifice mixer. The pressure is generally 10 pounds using nine or ten plates.

Ammonia is usually used in aqueous solution, while caustic soda of 10° Baume and of 0·2 to 0·3 per cent. by volume of oil is used. In the use of caustic soda deposition of the reagent and of sodium sulphide may cause local pitting while the rate of coke deposit in the heating tubes is increased.

Soda ash is cheaper than the hydroxide and causes less deposition. It is used as 10° Baume solution and 25 per cent. more by weight than caustic soda is required.

Lime is used to overcome the caustic embrittlement due to secondary corrosion in the use of caustic alkalis. Its success has been marked. From 0·4–1·0 pounds of slaked lime per barrel of highly corrosive charging stock is necessary, and the dry lime is mixed with a portion of the charging stock before injection into the system. Thus it is found that the heating tubes are more easily cleaned, due probably to a protective covering between the steel and coke, the coke is of better quality and more easily broken and the operation is cheaper than with caustic soda.

An analysis of the coke produced in a cracking unit with and without the use of lime :—

Quantity of lime used ..	0·4 lbs. per barrel ..	nil
Moisture	2·73 ..	3·23
Volatile matter	11·85 ..	13·28
Fixed carbon	78·71 ..	81·94
Ash (inclusive of lime) ..	6·71 ..	1·55
Lime	3·97 ..	—
Sulphur	1·23 ..	0·77
B.Th.U.	14,252 ..	15,219

A. R. S.

85. New High Strength Joint for Bronze-welded Cast-Iron Pipe. W. I. Gaston. *Oil Age*,, xxiv, 12.

A further series of tests has been carried out on the mechanical strength of various types of bronze-welded joints on cast-iron pipes. The first tests made in this connection indicated that the collar type of joint was stronger than the V, and the former type passed into general use. It was also known that the fracture in a collar type joint occurred in the iron near the weld, and the idea sprang up that the weld was stronger than the pipe. This is not true, a welded pipe having a mechanical strength less than 50 per cent. of that of the unwelded pipe. It was next thought that fracture occurred in the iron on account of physical change induced by the bronze, or from shrinking of the collar. This is also not the case, and the real reason for the weakness is the concentration of stress in the iron due to the shape of the joint.

The V-type of joint owed its initial failure to the bad mechanical contact between the bronze and the iron. This has been overcome by annealing the pipe after machining. It can then be tinned easily, and the V-joint made in this way has a mechanical strength about 80 per cent. of the plain pipe.

The type of fracture occurring with a V-joint suggested that a small surface of shear would be an improvement. This was tried, and the shear V-type of weld which is described in detail has a mechanical strength of nearly 100 per cent.

As regards expense, the shear-V costs slightly less than the collar, while the plain V weld is about 25—50 per cent. cheaper.

Tests show that corrosion is no worse at the bronze weld than over the whole pipe surface. P. D.

86. New British Short-Flame Burner for Pulverized Fuel. *Chem. and Ind.*, 46—52, 1222.

One of the disadvantages of the otherwise highly efficient method of pulverized fuel firing is the great length of flame, *e.g.*, 30 feet in a large water tube boiler. The new British short-flame burner enables the combustion to take place with a 10-foot flame. A single burner will take up to 150,000,000 B. Th. U., 5—6 tons of coal per hour, reducing the number of burners necessary from 5 to 1. 15 per cent. of the total air at 2 ins. W. G. pressure is supplied with all the coal directly round the centre of the burner by means of a concentric casing, while the remaining 85 per cent. air enters at 1 in. W. G. pressure with a violent cyclonic motion by means of another concentric case. Helical veins in the primary air-fuel casing prevent the separation of air and coal particles at the burner tip. A. R. S.

87. New Oil Engine Combustion Process. *Gas and Oil Power*, 1927, 23, 265.

This article is an extract from the report of investigations made by Professor Stribeck into the process of combustion in an engine developed by the Acro Company, under whose patents the Bosche Company is licensed.

The principle applies to engines of the solid injection Diesel type, and investigations were carried out on an ordinary Diesel engine fitted with special Acro-Bosch equivalents in place of the standard piston head, piston, injection pump and injection nozzle or valve. The test engine had a total displacement of 134·7 cu. ins., with a compression space of 8·54 cu. ins. The compression space may be divided into three sections, the space between piston and cylinder head occupying 23 per cent. of the total volume, a cavity

in the piston occupying 70 per cent. and a funnel shaped connecting passage between the two occupying 7 per cent.

The funnel shaped passage is referred to as the venturi and tapers towards the air chamber in the piston, the area of the opening into the air chamber is 0·181 sq. ins., and this opening is called the orifice.

Tests were made to determine pressure differences across the venturi and showed a maximum difference at 20° ahead of dead centre which subsequently decreased steadily but with very pronounced oscillations at or near top dead centre.

Temperature tests were made with thermo-couples of various lengths, some of which projected through the venturi into the air chamber, and curves are given showing variation of temperature with crank angle on the power stroke at eight different points.

From a comparison of results obtained it is found that, disregarding an incidental disturbance before 0·08 ins. piston travel, no combustion takes place in the air chamber, but that this chamber supplies air throughout combustion to a flame burning after the manner of a blow torch in the venturi.

It is stated that fuel and air are found to burn as if mixed in the correct proportions and that small quantities of fuel are burned as completely as larger quantities. E. R. B. S.

88. High-Speed Compression-Ignition Engine Research. H. B. Taylor. *Automobile Engineer*, 17, 235.

The relative merits of compression-ignition and petrol engines are discussed together with various systems of injection. For aircraft research a single cylinder engine was used, 8 ins. bore, 11 ins. stroke, working on the four-stroke cycle at 1,250 r.p.m. and 12 : 1 compression ratio. The fuel was delivered into a receiver by means of a pump controlled on the suction valve, the injection valve is positively operated by a cam and closed with a spring. No by-pass relief valve was fitted, the pump being the metering device. The time of injection was variable while the engine was running, but the period was fixed. Shale oil, sp. gr. ·86, was used as fuel throughout the tests. The spray nozzles used had, situated below the valve seat, a small hemispherical chamber out of which led several small holes. It was found that the best sprayer was one consisting of five holes, ·0145 in. diameter, ·02 in. long, drilled at a cone angle of 120°. Data are given for performance at various cylinder pressures. It is estimated that a suitable compression-ignition engine would weigh between 1 and 2 lbs. B. H. P. more than the corresponding petrol engine. Curves are shown demonstrating that owing to the reduced fuel consumption of the former engine an advantage is gained by using this type for long aircraft flights as compared with the petrol engine. W. N. H.

89. An Automatically Operating Device for Giving Warning of Sudden Changes of Temperature and of Predetermined Fixed Temperatures. A. Albert. E.P. 271,072.

This device consists essentially of three parallel rods fixed in a vertical plane and held at one end. They are allowed to expand freely under the action of the temperature they are designed to control. For the purpose of giving warning when a certain temperature is reached, an electrical contact is made by a point on the moving end of the middle arm, which on expansion to a certain extent, touches a fixed contact. The circuit so made, actuates a relay, and hence some audible or visible form of warning. For the purpose of giving warning of a temperature rise which is dangerously rapid, advantage

is taken of the fact that a thick rod will not expand as quickly as a thin one. The central rod is made thicker than the side rods, and carries a fulcrum at its free end on which a horizontal lever is pivoted. One end of this lever is fixed to the left-hand rod, its length being such that it overhands the right-hand rod. Under a rapid rise, the two outside rods expand faster than the central one, and the free end of the lever makes contact with the top of the right-hand rod. By this means, an electrical circuit is made as before, and an audible or visible warning is brought into action. P. D.

90. Wet Vacuum Pumping Apparatus. Marks and Clerk for I. C. Jennings. E.P. 280, 779. Nov. 24, 1927.

The object of the invention is to improve the wet vacuum pumping apparatus described in Patent Specification No. 192,365. The patent referred to describes an apparatus consisting of a rigid base, a tank secured thereto, an air pump and a water pump secured to the side of the tank and a motor for driving the pumps mounted on the base.

The mounting for the motor is improved by securing the motor directly to the pump casing, thereby assuring that the motor and pumps are in correct alignment and simplifying erection.

The invention further consists in improving details whereby the apparatus is provided with a float control for the electric motor. E. R. B. S.

91. Improvements in Meter Devices for Indicating the Viscosity of Fluids. Metropolitan-Vickers Electrical Co., Ltd. E.P. 272,251, Sept. 29, 1927.

The invention relates to a meter device for indicating the viscosity of a fluid comprising a rotatable drum adapted to be driven by a suitable motor; a viscosimeter drum located co-axially with and inside the rotatable drum, a pump of the impeller type carried by the rotatable drum, for circulating the fluid into the annular space between the drums, means for resisting the torque which is transmitted from the rotatable drum through the fluid to the viscosimeter drum and a device for indicating said torque. S. W. C.

92. Filter for Gases. M. C. E. Mulot. E.P. 279,000; Oct. 20, 1927.

A cylindrical wick is mounted vertically, the bottom dipping into a reservoir containing light lubricating oil. A tall bell cap completely covers the projecting portion of the wick. Air enters through a series of holes situated at the base of the bell, it passes up the outside of the wick, over the top and down the inside. Inside the wick cylinder is situated an Archimedian screw which gives the air a centrifugal motion bringing it into turbulent contact with the wick. Thence it passes to the engine. Any dirt which drops off the wick is collected in two circular cups situated one close round the outside and the other round the inside of the bottom of the projecting portion of the wick. W. N. H.

93. Improvements Relating to Radiation Pyrometers. A. Schwartz. E.P. 280,454.

The improvements relate to the type of radiation thermometer which is actuated by the expansion of a bi-metallic spring. Rises in temperature are communicated to the spring by the concentration on it of a beam of heat rays. This determines the extent of expansion of the spring. Owing however to the temperature of the surroundings of the pyrometer, the zero of such an instrument in its unmodified form, is not constant. The proposed alteration is designed to compensate for the alteration in the surrounding temperature by means of a second bi-metallic spring. The latter moves the circular

scale on which the pointer indicates the temperature and is shielded from the beam of rays. As the room temperature varies, and causes the pointer to alter its zero, so the second spiral causes the scale to follow the pointer. The movement of the pointer as indicated by the scale is then due wholly to the beam of heat projected on it. The scale is illuminated by light radiated from the hot body by means of a circular glass window which surrounds the object glass. P. D.

See also Abstracts Nos. 3, 5, 8, 20, 24, 25, 37, 107, 127, 128, 129, 134, 136 and 138, and those under the heading of "Refinery Plant."

Antidetonation.

94. A Study of Auto-Ignition Temperatures. H. J. Masson and W. F. Hamilton. *Ind. Eng. Chem.*, 1927, 19, 12, 1,335–1,338.

The apparatus used by the authors consisted of a well-lagged cylindrical electric resistance furnace, which constituted the heating element. The heated surface consisted of a circular platinum plate with an indentation at the centre to which was welded the hot junction of a Pt–Pt 10 per cent. Rh. thermocouple, the cold junction being set in a thermos flask fitted with ice. Temperatures were read on a very sensitive millivoltmeter registering between 650° F. and 1,400° F. Heavy Pt wires covered with Impervite insulators were welded to the edges of the plate and alternating current was used as a means of superheating the plate. A length of nickel tubing entered from the base of the furnace and coiled around, ending just above the edge of the plate. It was used to deliver air to the surface and remove the products of combustion. The air passed in was dried by means of sulphuric acid. A demountable aluminium cover was placed on the top of the furnace and supported a gravity hook-tube carrying a magnifying glass focussed on the plate and in the centre another gravity tube which served as an opening for the introduction of the drops. Drops of constant size were obtained by gravity flow from an ordinary hypodermic needle, the barrel of which served as a reservoir for the liquid, while drops of various sizes were obtained by the use of various sized needles. The furnace temperature is slowly raised and a drop of the liquid allowed to fall into the depression in the plate at regular intervals until a flash is seen within one second after the drop makes contact with the surface. The furnace temperature is now lowered 10° F. and kept there, and the alternating current through the plate is switched on, the temperature of the plate slowly raised till ignition takes place. The plate is then raised to 16—20° F. above this value, and the temperature slowly lowered until ignition takes place. By repetition the final ignition temperature is determined to the nearest degree. The results differ considerably from those of other observers, being in general higher, although the same general sequence of increasing ignition temperatures is maintained. The ignition temperatures of pure substances were studied, but no regular or periodic relationship was found connecting ignition temperatures and molecular weight, specific gravity, boiling-point, heat of combustion, heat of formation, vapour pressure, etc. It seems that there are two possible courses for the reaction, resulting in a race between hydroxylation and thermal decomposition. S. W. C.

95. Fuel for Internal Combustion Engines. Continental Aktiegesellschaft für Chemie. E.P. 267,079, Nov. 14, 1927.

The patentees trace the cause of engine knock to a state of electrical tension existing in the explosive mixture. They propose to eliminate this

tension by dissolving inorganic salts of heavy metals, such as lead nitrate, copper nitrate, carbonates of heavy metals, etc. This is achieved by employing suitable solvents. In an example, 5 kilograms copper nitrate is dissolved in 1 kilogram of methyl cyclohenanol made soluble in water by means of soap. This solution is filtered and mixed with 100 kilograms petrol. A small quantity of yellow liquor settles out and is withdrawn. W. N. H.

See also Abstracts Nos. 23 and 88.

Cracking.

96. Some Facts Concerning Vapor Phase Cracking. L. de Florez. *Refiner*, 1928, **1**, 71—75.

Liquid phase and vapour phase cracking may be designated respectively as low and high temperature cracking, 950° F. (510° C.) being the temperature of demarcation between the two processes. Liquid phase cracking developed by the desire to produce a gasoline resembling that obtained by normal distillation, by the fact that no commercial advantage existed in manufacturing a high temperature gasoline and because of the large make of gas which hitherto could only be used as plant fuel. Vapour phase cracking may be practised at temperatures as high as 1,400° F. (760° C.), yielding from 23 per cent. to 30 per cent. of gasoline in one pass, the gasoline being somewhat difficult to handle but possessing anti-knock properties of a high order. The gas may be utilised in the manufacture of solvents or it may find an outlet as an enriching agent for towns' gas. The chief problem encountered in vapour phase cracking is that relating to efficient heat transfer: the only method is to pass the gases through comparatively small tubes at a high velocity and to impart heat to them while flowing in a highly turbulent stream. Accurate temperature control is also of the highest importance. An account of the plant built for the New England Oil Refining Co. at Fall River, Mass., is given. The cracking tubes consist of one inch (I.D.) Shelby Seamless tubing bent in the form of a hairpin, suspended vertically in a chamber which is separated from the furnace by a cross-wall. The tubes, which are thus heated by the products of combustion, are capable of being removed from the top and a tube may be removed for cleaning or repair in three minutes. The plant comprises 8 units, each of a capacity of about 125 barrels per day. Over a period of some months, the average throughput was 830 barrels per day, with an idle time of 17 per cent. for shut downs for all purposes. The yield of N.E.P. gasoline varied from 23 per cent. with a high sulphur Mexican distillate to about 30 per cent. with a Mid-Continent distillate. The operating costs at full capacity were approximately 25 cents per barrel of charge, plus 20 cents per barrel for treating and re-running. The plant, which was erected in 1922 at a cost of 50,000 dollars paid for itself in five months. The average run was 7 days and the longest 17 days. Units built later for the Petroleum Chemical Company were similar in type and operated on a 30-day cycle, the longest run being 47 days.

The charge must be an overhead distillate and preferably it should be introduced as liquid to a series coil where it is vaporised and cracked. If charged to the cracking coils as vapours from a still, the speed of the vapours is insufficient to give turbulent flow. With proper control of temperature, practically no coke is deposited. The gasoline is yellow and has a characteristic odour. When treated with acid, it acquires a deep wine colour and must be re-distilled. Refining can be accomplished with acid or, in the

vapour state, with fullers earth. The losses are higher than with liquid-phase-cracked gasoline and the gum substances are present in larger quantities. Stability is not high and it is advantageous to blend at once with a straight-run product. H. G. S.

Cracking and Refining.

97. Cracking of Heavy Mineral Oils with Japan Earth. Kyuhey Kobayashi and Ken-i-chi Yamamoto. *Jour. Soc. Chem. Ind.*, Jap. **30**, 17B (1927); *Brenn.-Chem.*, 8.20, 329.

For this investigation of the cracking action of Japanese earth (acid clay) upon heavy mineral oils, illuminating oil, California heavy oil and California lubricating oil were used. The work was done with special reference to the yield of gasoline. Varying quantities of the oil were used in an electrically heated iron bomb. The use of Japanese earth had the same effect as higher pressure. The complicated action of the earth is probably adsorption, polymerization, condensation, and decomposition combined. The gasoline yield is directly proportional to the quantity of earth used. The heavy oils rich in paraffins crack most readily. W. H. T.

98. Products obtained from the Cracking of Lignite Tar Oil. J. Varga and A. Erdely. *Brenn. Chem.*, 1927, T.8, 9, 133–136.

In previous work, the authors have studied the influence of temperature, pressure and time on the cracking of lignite tar oils. In order to increase the yield of spirit, they have endeavoured to crack the residues from the first cracking. The refined lignite oils yielded as much as 47 per cent. of spirit and 31 per cent. of heavy oil. These spirits do not shew any essential differences from those obtained in cracking petroleum oils. Working at 10–20 atmospheres, the lignite oil commences to decompose between 370° and 390° C., the yield of spirit being a maximum at 420° C. At higher temperatures the yield of coke and gas is considerably increased. The spirit obtained from petroleum or lignite contains unsaturated compounds soluble in 100 per cent. sulphuric acid, and boiling between 180° and 190° C. The products soluble in caustic soda distil between 210° and 220° C. W. H. T.

99. Method and Apparatus for Cracking Oils. W. Brink. U.S.P. 1,652,344, 1927. Appl. March 3, 1925.

A series of cracking chambers is placed in communication with the lower half of a common still, which is vertical and unheated, and also with a drum surmounting the still. The oil to be treated enters the cracking chambers, passes thence to the drum and comes into contact with a baffle plate which causes the vapours to pass away through dephlegmators and the unchanged or partially cracked liquid to fall into the still. The refluxed oil thus falls slowly through the still, at the bottom of which carbon is deposited in a finely divided state. The oil then passes through the cracking chambers back to the drum and baffle plate. H. G. S.

100. Conversion Apparatus for Refining Hydrocarbon Materials. R. K. Collins. U.S.P. 1,654,577, 3.1.28. Appl. May 5, 1922.

The invention consists of a novel expanding and converting apparatus, designed to treat vapours according to the chemical, thermodynamic and mechanical laws of physical conversion. It provides an expander adapted to receive relatively large quantities of vapours for uniform heat treatment in order to convert and release their constituent parts.

A back trap converter is also provided to treat the heavier fractions of condensed hydrocarbons to revaporise and refine the same.

The expander is a vertical cylindrical shaped casing with a vapour inlet adjacent to the bottom. This casing is mounted on a conical shaped housing which provides a heat chamber and likewise supports the back trap converter.

Within, the expander tubes are supported vertically by top and bottom plates and perforated nickel plates are provided within the chamber to act as catalytic agents.

The back trap converter is a cylindrical casing mounted transversally to the housing of the expander. This converter is adapted to receive the condensate from a dephlegmator attached to the expander and superheated steam and permanent gases may be passed into this converter as part of the process of increasing the production of light products. The vapours from the back trap converter pass into the bottom part of the expander vessel and mix with the vapours therein.

No mechanical scrapers or coke removers are required in this apparatus as total decomposition of the hydrocarbons never takes place except at the will of the operator. R. P.

101. Expansion Process for Refining Hydrocarbon Materials. R. K. Collins. U.S.P. 1654578. Jan. 3, 1928. Appl. May 23, 1922.

The process consists of distilling a hydrocarbon in a horizontal still to provide a vapour which is led into a vertical expansion chamber wherein it is heated to a conversion temperature to release the constituents, and passing thence through a dephlegmator to separate the light fraction from the heavy fraction. The heavy fraction flows downwards into a re-run chamber heated by the flue gases from the still furnace, with superheated steam and permanent hydrocarbon gases, formed in the process.

The vapours produced are passed into the expansion chamber to be mixed with the vapours from the still. The process of conversion continues within the expander, in conjunction with the hydrogenation brought about by the steam and fixed gases introduced into the process. The breaking down of the hydrocarbons being treated within the expander, into fixed gas and carbon, is thus prevented and the formation of the desired products is facilitated. The reactions within the expander are such as to produce a maximum of the desired products. R. P.

102. Expansion Process and Apparatus for Refining of Hydrocarbons. R. K. Collins. U.S.P. 1,654,580. Jan. 3, 1928. Appl. July 28, 1926.

It is claimed for this process that it provides an improved means of producing anti-knock motor fuel by the vapour phase method. The improved treatment is carried out in a chamber free from any excessive pressure and the vapours are heat treated uniformly by means of uniformly spaced heating elements intercepting the area through which they pass.

The plant consists of a still for vaporising the oil, and an expander into which the vapours pass. The remainder of the plant is made up of conventional dephlegmators condensers etc.

The expander is a vertical cylindrical vessel, having headers at top and bottom, vertical tubes connect the top and bottom headers, and perforated baffle plates are provided through which the vertical tubes pass. Flue gases from the still pass up through the vertical tubes which serve as the heating elements. The vapours from the still pass into the shell of the expander and surround the heating tubes, flow upwards and leave at the top. The perforated baffle plates assist in distributing the vapours, which may be all vapour or partly vapour and partly steam.

By properly controlling the rate of feed and the temperature of the expander the amount of carbon formed can be reduced to zero. Both the yield of gasoline and the percentage of unsaturated compounds are controlled independently of each other by changing the heat imparted, the quantity of steam supplied and by changing the rate of vapour feed. R. P.

103. Method for Treating Petroleum Oil A. D. David. U.S.P. 1,655,596. Jan. 10, 1928. Appl. Feb. 14,. 1923.

The process is a pressure distillation process for the production of cracked motor fuel. The plant consists of a heating coil, two vaporising chambers, a dephlegmator, condenser and receiver.

The oil is introduced and raised to cracking temperature in the heating coil, and directed into either of the expansion chambers. When the carbon in the first chamber has become objectionable this chamber is cut out and the second is utilized while the first is cleared of the accumulated residual carbon or coke. As a safety measure, means are provided for keeping the valves and pipe line, leading to either expansion chamber, cool, by circulating cold oil under an adequate pressure whilst the chamber is in process of cleaning.. H. G. S.

104. Process for Converting Oil. W. M. Cross. U.S.P. 1,644,991, 1927. Appl. April 3rd, 1923.

The oil is heated to cracking temperature in a tubular preheater and then transferred to an elongated insulated reaction chamber, sufficient pressure, *e.g.*, 600 to 1,000 pounds per square inch being maintained to prevent any substantial vaporisation of the oil. The latter then enters a vaporising still or column at approximately atmospheric pressure. The run-back from the still is withdrawn continuously, whilst the vapours pass to a second still or column, the vapours from which constitute gasoline and the run-back recycling stock. Means are provided to maintain a constant level of liquid in each of the two stills. H. G. S.

105. Process for Converting Petroleum Oils. C. P. Dubbs. U.S.P. 1,652,166, 1927.

The invention is designed simultaneously to dehydrate and crack petroleum. The raw stock is taken through a closed pipe built into a dephlegmator and then into an enlarged zone from which the water distils away. The dry preheated oil now joins the reflux from the dephlegmator, traverses a cracking coil and enters an expansion chamber from which the heavy residues are withdrawn and the vapours pass forward to the dephlegmator and ultimately to coolers, condensers and receivers. H. G. S.

106. Treating Emulsified Oils. G. Egloff and H. P. Benner. Assrs. to Universal Oil Products Co., Illinois. U.S.P. 1,649,102. Sept. 18th, 1920.

A process is described of simultaneously dehydrating and cracking emulsified petroleum oils in such a way as to prevent frothing and also to convert a substantial portion of the oil content into gasoline, kerosene or similar light hydrocarbons.

The emulsified oil is subjected to surface heating to dehydrate it, then heat is applied to the lower portion of the oil to further distil and crack it, the generated vapours being further superheated to crack them, meanwhile maintaining a substantial pressure on the oil under treatment and continuing the process until the oil has been dehydrated and a substantial portion thereof converted to lower boiling point hydrocarbons. H. G. S.

107. Apparatus for Cracking Oil. G. Egloff and H. P. Benner. U.S.P. 1,649,105, 1927. Appl. Feb. 11th, 1921.

The invention relates to the construction of the transfer line connecting the heating coils and the expansion chamber of a cracking plant. The discharge end of the transfer line terminates in a number of internally rifled nozzles so that the oil emerges at a high velocity in a plurality of circulatory paths. Directly in front of the nozzle is placed an impact or baffle plate against which the jets of oil are projected. By this means, operating on Healdton, Oklahoma fuel oil of sp. g. 0·891 under a pressure of 200 lbs. per square inch and a temperature of 895° F. (480° C.) in the transfer line, 35 per cent. of gasoline of sp. g. 0·744 was obtained. H. G. S.

108. Apparatus for Treating Hydrocarbons. G. Egloff and H. P. Benner. U.S.P. 1,655,600. Jan. 10th, 1928. Appl. June 28th, 1920.

An apparatus is specified for cracking oils, in which a constantly rotating or agitating device is used. It consists of a still with a central heating flue. Between the heating flue and the shell of the still a rotating member, provided with a number of paddles, constantly agitates the oil. Means are provided for feeding the oil into the space between the member and the heating tube and a vapour discharge pipe is connected to the still.

The arrangement is such that all portions of the oil are subjected to uniform treatment and avoids the production of "hot spots" and dangerous carbon formation.

The apparatus is so constructed that the inner shell can be readily cleaned.

It is stated that about 70 per cent. of pressure distillate may be obtained containing 65 per cent. or more of gasoline. R. P.

109. Treating Oil with Aluminium Chloride. F. W. Hall. U.S.P. 1,647,445, 1927. Appl. Mar. 15, 1920.

The process and apparatus are devised, either to prepare a mixture or solution of aluminium chloride and oil suitable for the refining of a bulk of oil, or for the direct conversion of oil in presence of aluminium chloride. The latter is prepared in any known manner and its vapours contacted with a counter current of oil, which may be heated if cracking is desired, or used at the ordinary temperature if the object is merely to obtain a solution. In the latter case, the two layers, one of oil and the other of oil and aluminium chloride, are separated and the former recirculated. H. G. S.

110. Apparatus for Effecting Chemical Reactions. H. Hennebutte and E. Goutal. U.S.P. 1,650,713. May 21, 1923.

The patent describes an apparatus for effecting chemical reactions particularly adapted for carrying out dehydration, distillation or generally speaking, any chemical reaction including (1) either pyrogenetic decomposition with or without the addition of reagents, (2) or oxidations, reductions, hydrogenations, sulphurations, etc.

The apparatus essentially comprises:—(1) a stirring device constituted by one or more endless screws, worms or translating propellors operated or driven from the inside and located in metal troughs which are simple or twin. These screws are provided with special devices to turn over the material and to submit the different parts to the reagent and to the action of heat. Screws are provided for this purpose with vanes or mould-boards extending from one coil to another, constituting a ladle or scoop. (2) Lids which cover the metal troughs and fit snugly upon the latter. These are provided with large exhaust openings and interiorly provided with gutters

for receiving the partially condensed products and conveying them outside. Each trough constitutes a member of a furnace heated to a determined temperature. The material to be treated is placed within these members through which it passes from one to the other, each being heated at different temperatures. During the operation it is possible to introduce into the members any solid, liquid or gaseous product, which is caused to react upon the matter under treatment, the resulting products being always exhausted in the same conditions. H. G. S.

111. Treating Hydrocarbon Oils. R. C. Holmes, F. T. Manley and O. Behimer. U.S.P. 1,646,380, 1927. Appl. 10.12.23.

The oil to be cracked is heated to conversion temperature and discharged into a battery of stills in which cracking conditions are maintained, the vapours being partially condensed and the condensate returned to the stills. The latter are provided with residue draw-off lines leading to a common header and with interconnecting vapour and liquid level lines. The arrangement of valves and lines is such that the pre-heated oil can be selectively introduced into one or more stills and the reflux from the bubble tower returned to any desired still, the object being to maintain the oil in the several converters at substantially the same boiling point. The operation is conducted under super-atmospheric pressure, which is preferably controlled mainly on the valve in the gas outlet. H. G. S.

112. Art of Distilling Oils. R. E. Humphreys. U.S.P. 1,647,629, 1927. Appl. 21.3.1922.

A heavy oil distillate is distilled under pressure and the pressure distillate so produced is topped to remove cracked gasoline. The pressure distillate bottoms, instead of being utilised as recycling stock, are subjected to a further distillation under a pressure exceeding 300 pounds per square inch. H. G. S.

113. Improved Method of and Installation for the Hydrogenation and Cracking of Hydrocarbon Compounds. Inter. Bergin-Comp. voor, Olie-en Kolench and A. Debo. E.P. 280,734, 1927.

The process is an improvement on the known method of hydrogenating coal, tars, oils and the like and is designed to utilise the energy contained in the compressed products and also to increase the yield of benzine. The products from the high-pressure reaction vessel are partially cooled and expanded from say, 150 atm. to 100 atm., into a pressure separator. The heavy products, ash, iron oxide, etc., are withdrawn and the vapours taken through a cooler to effect condensation of an intermediate oil fraction, which is then employed to scrub the remaining gases and vapours, whilst still under a pressure of, say, 100 atm. The residual, scrubbed gas is finally heated and discharged to an engine which takes up the remaining pressure. H. G. S.

114. Art of Cracking Hydrocarbons. E. W. Isom. U.S.P. 1,650,519, 1927. Appl. 28.1.22.

In an apparatus comprising heating oils, pressure still, reflux tower, condenser and gas-holder, means are provided to charge the cracking stock into the top, and gaseous hydrocarbons from the gas-holder into the bottom, of the reflux tower. The mixture of reflux and raw stock traverses a pipe passing through the bottom of the still and after being raised to cracking temperature in the coils, is returned to the still near the bottom thereof. H. G. S.

115. Treating Hydrocarbon Liquids. R. C. Kirwin. U.S.P. 1,654,654. January 3, 1928.

An improved apparatus and method for cracking heavy hydrocarbons such as crude oil or kerosene to produce a greater quantity of the more valuable light hydrocarbons, without the necessity of using pressure. More effective temperature control during the cracking operation and prevention of formation and precipitation of carbon in the cracking zone is claimed. Crude oil vapours are directed from a crude oil still through a vapour line and an electrically heated conduit to a condenser. The conduit is horizontally disposed so that vapours under low pressure will more readily flow therethrough. The condensate passes into a receiving tank from which it is pumped to a steam still where gasoline vapours are formed, and pass through a condenser to a tank. During the early stages of operation the residue in the steam still is kerosene. This kerosene is returned to the crude still for further vaporisation under increased temperature, and again passes through the system as described. In the final stages a residue is left in the steam still not capable of further vaporisation in the crude still. This is drawn off and may be used as fuel oil. The pressure in all parts of the system is preferably never at any time greatly in excess of atmospheric pressure. Within the conduit above mentioned are electric heaters consisting of a refractory core wound with resistance wire, with terminals connected to a suitable source of electrical power. An insulating cover of mica surrounds the wire and a copper cylinder surrounds the mica, together forming a protective covering against direct contact of the wire with vapours and preventing carbon formation thereon and overheating of the vapours. The conduit consists of an iron, steel or copper pipe, adjacent to each end of which is a deflector so formed as to direct the vapours into contact with the heaters. Satisfactory results are obtained with the temperature of the vapours passing through the conduit only 25° F. to 50° F. above the temperature within the crude still. Temperature in the conduit is controlled by the showing of tests of the liquid in the receiving house. H. G. S.

116. Apparatus for Treating Hydrocarbon and Other Materials. D. E. de Lape. U.S.P. 1,652,563, 1927. Appl., May 7, 1921.

The oil to be cracked is introduced into the bottom of a chamber which is filled with molten metal and with pieces of metal having a higher melting point than that of the molten body. The oil, which may be mixed with steam, is made to take an extended passage by means of baffles, the latter being individually heated—*e.g.*, electrically. The treated oil then enters a vapour chamber from which vapours and residue are separately withdrawn. The operation may be conducted under pressure. H. G. S.

117. Process of Carrying Out Chemical Reactions with Liquids. E. H. Leslie and B. R. Tunison. U.S.P. 1,644,736, 1927. Appl. Sept. 4, 1920.

The process consists in cracking oil by bringing together surfaces of liquid hydrocarbons at a converting temperature as high as the boiling-point of one of them. This is effected by distilling a heavy oil, condensing the heavier evolved vapours and causing the condensate to be uniformly distributed over the surface of the main body of oil in the form of drops having a diameter of at least one thirty-second of an inch. H. G. S.

118. Method of and Apparatus for Treating Petroleum. J. W. Lewis, junr. U.S.P. 1,636,520, 1927. Appl., April 4, 1925.

The apparatus consists essentially of tubes in which the oil is heated to conversion temperature in the vapour phase and a reaction chamber in which

the major portion of the cracking occurs. The tubes are fitted with a stationary helix of such dimensions that the cross-section of the helical path is a small fraction of the total cross-section of the tube. H. G. S.

119. Refining of Petroleum Oils. P. McMichael. U.S. Pat. 1,655,068, Jan. 3, 1928; Appl. Oct. 23, 1923; and 1,655,069, Jan. 3, 1928; Appl. Feb. 14, 1924.

Petroleum distillates, particularly those obtained by cracking, contain certain terpenes, diolefins, or isomers and polymers, and other homologues or derivatives which possess the property of forming gums or resins. These compounds are particularly objectionable when brought into contact with hot metal surfaces such as occurs in the internal combustion engine. Also oils containing sulphur either free or compound are disagreeable in odour and undesirable in petroleum distillates.

By means of this process, these objectionable features of cracked motor fuel are removed.

The process consist of digesting the oil with lime or lime and an ammonium salt under a reflux condenser for a specified time, thereafter treating with 75/79 per cent. of sulphuric acid, then treating with acid of greater concentration than 83 per cent. and finally distilling the product with steam.

By this method it is claimed that Mexican straight run distillate with 1.56 per cent. of sulphur can be refined to yield a product containing only ·01 per cent. sulphur. R. P.

120. Oil Still. J. Primrose. Asst. to Power Specialty Co., New York. U.S.P. 1,654,347. Dec. 27, 1927.

An improved form of cracking still is described having amongst other advantages, (1) a better and more even distribution of the hot gases over the entire chamber containing the oil pipes and an improved means of regulating the temperature and securing a more complete combustion of the fuel; (2) a more effective flow of oil through the system thereby securing better temperature control; (3) a more ready means of inspecting the tubes; (4) a more convenient means of withdrawing an impaired tube section for replacement by a new section.

The still is in the main of usual construction with a furnace or combustion chamber in front, a bridge wall and a bank of oil pipes arranged at the back of same. The source of heat may be an oil flame or coal fire, and the hot gases pass up over the wall and down through the bank of pipes and off to the flue through an opening controlled by a damper. An excess of cool air is drawn up through the bottom of the furnace, which is a chequered partition or grate beneath which is a chamber with a number of controllable dampers, and secures more perfect combustion. The air is mixed throughly and uniformly with the hot gases, and the temperature of the mixture may be closely regulated at all times by adjustment of the dampers.

The oil pipes are arranged in sections containing two horizontal rows of pipes, each section separated by a space. The chamber containing the pipes is provided with parallel sight holes extending vertically over the entire bank of tubes and normally closed by doors. By this means pipes may be examined or removed.

The pipes in each vertical staggered row are in series, while all the series connect with upper and lower manifolds. Oil is introduced at the bottom and passes upward, thus securing a perfect flow counter to that of the hot gases, which pass downward.

The oil pipes are enclosed in corrugated cast iron envelopes, the thickness of which vary with the temperature of the gases to which the pipes are exposed

so as to secure an even temperature in the oil as it issues from the pipes. Each vertical series of pipes is provided with a thermometer at a point just before it delivers into the outlet manifold, and by the adjustment of valves just beyond the inlet manifold the temperature may be regulated by the rate of flow of the oil. The ends of each pipe section are provided with rectangular cast iron flanges against which the corrugated envelopes abut. The lower pipe sections of each group rest upon I-beams in the setting, so that the upper sections are firmly supported by the flanges. The walls of the heating or pipe chamber are built up of solid blocks or bricks of refractory material capable of ready removal to facilitate replacement of a defective pipe section H. G. S.

121. Treating Hydrocarbons. U.S.P. 1,655,741. A. Schwarz, Jan. 10, 1928. Appl. Feb. 13, 1923.

Heavy fuel oil is distilled from a still and light gas oil from another still. The vapours are combined in a mixing chamber and pass into a condenser and receiving tank from which the gases may be recycled. The vapours from the heavy oil still are mainly unsaturated, and the vapours from the gas oil still are mainly saturated. By vaporising both and mixing in the mixing chamber along with the gases produced in the process, a rearrangement of molecules is produced.

It is immaterial whether the vapours get their reaction temperature during the period downward from heating to cooling or upward from hot to cold as long as they are passed through a series of heat conditions and at one stage or another are subjected to the proper conditions for conversion.

It is suggested that when a series of hydrocarbons ranging from asphalt to gas is heat-treated, the hydrocarbons of lower hydrogen content tend to combine with the hydrocarbons with higher hydrogen content, which in turn tend to combine with the hydrocarbons of the next higher series, and so on, provided the proper proportions of saturated and unsaturated hydrocarbons exist at each step. R. P.

122. Continuously Cracking and Fractionating Hydrocarbons. A. C. Spencer, Assr. to Standard Development Co., Delaware. U.S.P. 1,648,967. July 3, 1923.

The invention consists of a process of continuously subjecting a stream of hydrocarbons to a cracking temperature, separating out fractions heavier than kerosene in a series of progressively cooler zones provided with a controlled reflux, then fractionally separating the remaining vapours into kerosene and gasoline by feeding the vapours about midway in a second series of progressively cooled zones provided with a controlled reflux. H. G. S.

123. Oil Cracking Apparatus. F. C. Vande Water and F. R. Sunderman. U.S.P. 1,655,030. Jan. 3, 1928. Appl. Aug. 29, 1922.

A furnace and stack are contained within a still having parallel sides and arched roof. Within the still, a bell-shaped section compels the oil to be cracked, to travel upwards within the still, then downwards and finally upwards towards the outlet.

The idea underlying the design of this apparatus is apparently to cause the oil to pass through a mass of heavy residuum at such a rate that the incoming oil does not materially reduce the temperature of the residual oil contained within the body of the still, but the incoming oil is immediately vaporised by the highly-heated residuum and the molecules of the hydrocarbons are decomposed or cracked. The resulting vapours pass into a condenser to a receiver. The period of contact may be varied by lowering or raising the bell-shaped member within the still. R. P.

124. Process of and Apparatus for Distilling Oil. G. W. Wallace. U.S.P. 1,650,169, 1927. Appl. Sept. 17, 1923.

The process consists in subjecting a stream of oil maintained under cracking conditions to the catalytic action of metals composing elements which move continuously through the oil stream. The cracking still is built up of a number of vertical tubes arranged in pairs and heated by suitable means, endless chains constructed of copper, cobalt, iron, nickel, platinum, magnesium or aluminium, etc., or carrying deposits such as titanium oxide, or the chlorides of aluminium, iron, calcium, etc., being made to pass in an upward direction through the first and downwardly through the second of each pair of pipes. The chains pass over sprockets arranged in the upper header of each section of pipes and under sprockets in the lower header, and may be so devised that they remove deposited carbon from the surface of the tubes. H. G. S.

125. Treating Hydrocarbon Oils. E. R. Wolcott. U.S.P. 1,654,711, January 3, 1928. Appl., October 1, 1924.

In this process the oil to be converted into lighter products is charged into a confined space and subjected to the disruptive action produced by an explosion or rapid combustion of rapidly burning material. The plant designed for the purpose of carrying out this process consists of a still within which a number of U-tubes are mounted. One arm of each tube projects through the shell of the still and is connected to a combustion chamber, provided with a sparking-plug or other means of ignition. Each combustion chamber has tubes to supply oil and a combustible gas. Air is also supplied or the proper combustible mixture of gas and air is admitted through one pipe. The other limb of the U-tube ending within the still is restricted in order to assist in producing the desired pressure. From the still vapours pass into a dephlegmator and condenser. In carrying out the process a charge of oil is admitted into the U-tubes. The explosive mixture is then charged and ignited by means of the sparking-plugs producing a high pressure and temperature sufficient to produce decomposition. The oil is then expanded through the restricted outlets into the still, and the vapours pass into the dephlegmator.

126. Process for Manufacturing Light Hydrocarbon Liquids. W. S. Yard and E. N. Percy. U.S.P. 1,643,401, 1927. Appl., March 17, 1924.

The process consists in spraying heavy oils upon the surface of a deep bed of incandescent solid fuel contained in a gas producer and passing the vapours and gases produced, together with steam, through a mass of finely divided carbonaceous material heated electrically to redness. H. G. S.

See also Abstracts Nos. 13, 18, 23, 26, 29, 34, 36, 60, 77, 142 and 143.

Oil Field Technology.

127. Equipment of Air-Gas Lift in Fields Outside the Seminole. R. W. Bond. *Oil and Gas J.*, 26, 23, 63.

The gas lift is a comparatively recent development in the Oklahoma and Kansas fields and during its initiation usually employed small tandem compressors of about 250 c. ft. per minute displacement, driven by automotive type gas engines with short belt drive, compressor units being placed at each well head. After the method had been proved successful it was considered advisable to centralise compressor equipment, but in view of the uncertainty

of information concerning "gas-lift flowing life" most of the installations were of the semi-portable type.

The electric motor came rapidly into use for small compressors as soon as electric power became available, as the greater continuity of service was considered to outweigh the increase in running costs.

Apart from semi-portable units a number of strictly portable compressors were used, consisting of automotive type gas engines directly connected to vertical compressors. The unit was mounted on one base with wheels or skids, but had a capacity of only about 150 c. ft. per minute.

Those operators who used non-portable units at the first centralisation installed large duplex compressors, belt-driven by slow speed gas engines. This type did not prove popular, and the present trend in large compressor equipment is the direct-connected duplex, driven by twin-cylinder gas engines of about 180 horse power. In addition to these there are at present in use a number of the smaller semi-portable direct-coupled units consisting of vertical tandem compressors and vertical slow-speed gas engines. These engines have interchangeable heads enabling oil fuel to be used as an alternative.

In operating the first installations, no attempt was made to control gas volume supplied to wells other than by the number of compressors used, and with the coming of the central compressor station this was at first left practically unaltered but the air lines were so arranged that it was possible to switch any compressor, or number of compressors, on to any well.

Now that close volume control is recognised as being of great importance, in gas-lift work the compressors are arranged to deliver into a common manifold in which a reasonably constant pressure is maintained. The general practice is to lead separate pressure lines from this manifold to each well and so concentrate all volume-controlling apparatus in the central station, at the expense of extra piping, rather than to run one high-pressure loop main round the area served with take-offs to each well and volume-control apparatus at the well head. With the manifold system the pressure maintained is slightly higher than that of the highest operating pressure of the wells, and arrangements are made so that when starting a well it may be coupled direct to a compressor instead of the manifold and so obtain the necessary extra pressure.

Attention is drawn to the importance of using connections sufficiently strong to withstand the very high starting pressures sometimes encountered.

Natural gas is used as the lifting medium in most cases outside the Seminole area, and as the gas flows in a continuous cycle with relatively small losses only a small volume of make-up gas is required.

The lay-out of pipe-lines at the head of the well has been found to have a considerable influence on efficiency. The flow line from control head to separator should be short and free from sharp or right-angle bends.

Three diagrammatic sketches are given showing suggested arrangements at the well head. E. R. B. S.

128. Safety Rod Head and Instant Action Circulating Head Developed. *Oil Age*, **24**, 10, 48.

The Loomis Oil Well Control Company, of Long Beach, have produced two articles of interest to those concerned in oil production.

The Loomis Safety Rod Head is constructed of cast steel and screws into the tubing head. It has a rubber insert which will strip clean all the rods as they are drawn out, at the same time allowing oil and gas to flow out through the lead line.

The other article is an instant action pressure seal head which may be used for stopping the flow of oil and gas when casing or tubing are being moved,

or as an instant change circulating head. The head is formed in two parts consisting of a body and a rubber insert in which are fixed a complete set of slips. The head is slipped on to the tubing and, when pressure builds up inside, the rubber contracts on the slips, firmly gripping and sealing off the tubing. One size of head will work satisfactorily on several sizes of tubing.
E. R. B. S.

129. Mechanical Apparatus for Air-gas Lift. J. L. Dwyer. *Oil and Gas J.*, 26, 83.

The article gives a short description of the apparatus used on an average lease in the Seminole Field. A sketch showing general lay-out and dimensions is included. The plant described is housed in three buildings, the engine-house, the control-house and auxiliary building. The engine-house contains four 90 h.p. gas engines driving four gas-compressors by means of a short belt with idler pulley. The main headers for gas-engine fuel, cooling water, and compressor suction and delivery are carried along the outside of the engine-house with suitable branches to each unit. A small high-pressure header is included for kick off when starting wells. The gas-delivery header, after delivering gas through a scrubber, leads the compressed gas to the control-house, which contains automatic volume-control apparatus on the branch lines leading off to each well. The gas passes through scrubbers before entering the suction header to the compressors.

The auxiliary building contains the electric-light plant and air-starting unit. The cooling water for both engines and compressors flows by gravity from the supply tank, through the cylinders and into a hot well dug outside the engine-house. A centrifugal pump delivers water from the hot well to the top of a cooling tower, from which it runs down to the main supply tank.

In most cases it is found that a working pressure of about 200 lbs. the square inch is suitable.

Since the buildings are usually only of a temporary nature, they are, as a rule, constructed of steel sheeting on a structural steel framework and are provided with floors made of chat.
E. R. B. S.

130. Relation of Air-gas Lift to Gas-oil Ratio and Bearing on Ultimate Production. F. W. Lake. *Oil and Gas J.*, 26, 22, 122.

In oil wells where gas is the main expulsive force, the ultimate production is inversely proportional to the gas-oil ratio. This will be understood by considering the conditions controlling recovery. Where oil and gas exist in a sand, and water movement is not freely possible, the force which drives the oil to the well is derived from the expansion of the gas. If, in the natural state, pressure and solution conditions are such that, if reduced to amospheric conditions, the ratio between gas and oil volumes is 100 to 1, and if in producing oil from the well 300 volumes of gas are delivered to one volume of oil, it is obvious that the expulsive force will be exhausted when only one-third of the available oil has been raised.

The only factor under control which influences the gas-oil ratio is the effective back pressure existing in the well opposite each oil-producing strata, and for any well at any particular time there is a back pressure which will produce a minimum gas-oil ratio. It is not always possible to apply the most-desirable back pressure, owing to practical considerations, and in such cases a compromise must be decided upon.

Owing to the number of variable factors available in the gas-lift which have a direct effect upon back pressure, it is usually possible to get a lower gas-oil ratio with this than with any other method of raising oil from non-

flowing wells. In a flowing well the back pressure may be varied by impeding the free escape of oil from the tubing or by pumping gas into the casing, as in the gas-lift practice.

In illustration of the effects of back pressure and gas-lift upon production and gas-oil ratio, records are given of the performance of several wells in South California. Curves showing average monthly yields of gas and oil and other particulars for two wells are given in *The Oil Age*. E. R. B. S.

131. Effect of "Pinching" on the Gas-oil Ratio. K. B. Nowells. *Oil and Gas J.*, 26, 28, 254.

While it is not possible to control the original energy determining the flow of a well, the operator may control the rate at which this energy is expended. As the energy is contained in the gas causing the pressure on a field, it is logical to regard the efficiency of a well as determined by the ratio of gas to oil produced. This paper describes experiments on wells in the Salt Creek field, carried out with the purpose of discovering the effect of various means of control on the gas-oil ratio.

Preliminary tests carried out on seven wells showed that, when the control was carried out by means of gate valves, the oil flow was reduced, but the gas-oil ratio was increased, that is, the efficiency was lowered.

The wells were produced as follows ;—

(1) Through casing with the flow controlled by means of (*a*) gate valve, (*b*) flow nipples in the flow line.

(2) Through plain tubing.

(3) Through tubing with oil flow controlled by : (*a*) gate valves, (*b*) flow nipples at the bottom.

(4) By "stop cocking."

(5) By means of flow devices at the bottom of the tubing.

(6) Through tubing and packer.

The results indicate that it is necessary to study a particular well carefully before deciding upon a method of control. Some wells give an increased efficiency under any method. Control on others, however carried out, brings a decrease in efficiency, and such inefficient wells should be closed in when possible. A third type of well responds to some methods of control, but not others.

Casing flow was easier to control by means of flow nipples rather than gate valves, and a better gas-oil ratio was obtained. Producing a well through tubing was usually the best arrangements. In general, methods which give small flowing pressures are the most efficient.

The work showed that pressure plotted against percentage flow gave straight lines for inefficient wells, and hyperbolas for efficient ones.

The figures obtained in the tests are recorded in tables and illustrated graphically. P. D.

132. Principles of Air-Gas Lift Practice. H. R. Pierce and James O. Lewis. *Oil and Gas J.* Vol. 26, No. 22, 42

Much has been written on the subject of air or gas-lift as applied in the oil field and certain claims have been based on incomplete or inaccurate data. This has led to a general element of doubt as to the reliability of a large part of the compiled data, and this article is a discussion of the sources of error and an explanation of some of the factors relating to the application of the air-lift principle to oil production. No distinction is made between air and gas-lift as they are generically the same.

The ends sought in applying the air-lift may be grouped in four main classes; to increase daily production to the greatest extent regardless of all else, to lift oil cheaply both as regards installation and operating costs, to provide an alternative method of pumping in very deep and crooked holes where a plunger pump can often not be worked and to increase oil recovery as distinct from oil production. It will be obvious that the design and operation of a gas-lift will depend upon the reason for its introduction and will differ with the standard of efficiency to be maintained.

The elements of the air-lift. may be gathered by considering a U-tube having one long and one short limb and a small air inlet pipe projecting up the longer limb a short distance from the bottom. Water is fed continuously into the short limb, which represents the submergence, air is injected continuously through the small branch pipe and what may be conveniently be considered as alternate small plugs of air and water rise continuously up the long limb or eduction, the air plugs expanding due to the fall in pressure as they rise. The water in the short limb will reach a certain level and remain constant so that its head just balances the friction in the tube and the pressure at the point of injection, similarly the weight of water in the flow tube combined with the friction in that tube will produce an almost equal pressure which will in turn be equal to the pressure in the air supply main less the friction in the air pipe. If the water supply is increased more air will be required to maintain the same submergance, or the air pressure must be raised. A decrease in water supply will require less air pressure, or less volume of air, until a point is reached where the air fails to lift and passes alone up the flow pipe, or water may collect in the flow and air pass back through the submergance tube. Similarly a decrease in air supply may stop the flow by allowing water to accumulate in the flow line and so produce excessive back pressure.

For a continuous air lift the necessary elements are a pressure that will feed the oil to be lifted against the lift pressure and an equal pressure to feed in the gas, the liquid pressure confining the gas and directing its flows upwards. The volume and pressure of gas must supply sufficient foot pounds of energy to lift the liquid, overcome friction and allow for losses caused by slippage or drop back of liquid in the flow tube. Air pressure is governed by the weight of liquid in the column and by friction, while volume is controlled by the amount of energy required at that pressure.

If the rock pressure falls below the air pressure necessary to lift oil the well must be worked intermittently in conjunction with a chamber at the well bottom, which may be artificial or, in tight sands, may be formed by the sands themselves. Then oil is allowed to flow into the chamber until full, when air pressure is applied both to the oil surface and the injection pipe thus forcing some oil up the well and some oil or air back into the sands.

The three main elements consuming energy are :—

(1) The weight of oil combined with distance lifted ; (2) the frictional resistance of oil and gas in the pipe ; and (3) the slippage or drop back of oil past the rising gas.

The first is obviously irreducible, as being the useful work done, while (2) and (3) give a field for endeavour to increase efficiency. Friction increases with velocity, but since there is a steady acceleration from the bottom to the top of the flow pipe due to expansion at reducing pressure, the calculation of pressure is an involved problem. To reduce friction velocity must be reduced, but it is found that slippage varies inversely with velocity so that it is necessary to strike a balance between the two requirements.

For any particular depth and quantity of fluid to be raised there is a certain diameter of tubing, pressure and volume of gas which gives the most efficient

working, but as a rule the diameter of tubing has to be fixed, or at any rate limited, by other practical considerations.

Tapered tubing offers one method for increasing efficiency by reducing the excessive velocity at the top of the well, but in deep wells, where it is especially desirable, the size of the casing limits the diameter at the top while a correctly proportioned tube would have a diameter at the bottom too small to pass a reasonable quantity of oil. In many cases it is found advisable to use plain tubing so long as a well is producing strongly and the large output will cover small losses in efficiency, but to instal tapered tubing when the output falls off and it is possible to use a smaller diameter of tube at the bottom of the well and increase the efficiency of the lift. It is pointed out that considerable advantages are realised in using tapered tubing if the casing of the well is made of a larger diameter near the surface, as the tapered flow tube can then be better applied.

The design of surface flow lines and the distance the oil has to travel before the gas is separated may have a considerable effect on efficiency. The air pipe can always be smaller than the flow pipe but in deep wells a surprisingly great drop in pressure may be caused by too small a pipe, and this point should be considered. Foot piece design has but small effect upon efficiency and the merits of different designs of air injectors has been much exaggerated.

There is much misunderstanding as to the use and significance of the "gas-oil ratio," or "gas factor" and it is explained that this ratio is only a measure of efficiency when all other conditions are the same. It is clear that the energy put into a well is a function not only of the volume but also of the pressure of gas supplied, so that general comparisons should be reduced to horse-power or to foot pounds of work done. Actually, however, since changes within the sand body do not as a rule take place very rapidly, the difference in efficiency of any single well may be measured by volume of gas alone, when varying the conditions in search of the best results, but it should be remembered that sufficient time must be given on any one set of conditions for pressures below ground to reach a steady value. The gas-oil ratio cannot fairly be used to compare the efficiencies of two different wells even though they be in the same field, as the pressure should also be taken into account and results given on a truly comparative basis. It should be remembered that efficiency in recovery is mostly governed by underground conditions.

As regards improvement in oil recovery by the use of the gas-lift it seems doubtful if there is any important effect other than back pressure applied to the sand, and it appears that there is an optimum pressure which will produce the best recovery in any well. This optimum pressure must often be departed from when it is required to lift the maximum possible volume of oil per day. There is as yet insufficient data to be able to work out the use of back pressure or to design an air-lift so as to get the back pressures desired and at the same time have an efficient air lift within the limits imposed by the size of the casing and other practical and economic conditions.

It is pointed out that the air lift may reduce the back pressure on the sand of a naturally flowing well, or even of a pumping well, in spite of the fact that several hundred pounds of air pressure are applied.

Tests on the deliveries of air compressors have indicated that figures based on manufacturers' claims and ratings are very unreliable. Similar errors are noted in other observational data and until information gathered in the field is more complete and dependable progress in air lift engineering will not be satisfactory. E. R. B. S.

Oilfields.

133. KEEPING GAS FROM FREEZING AT WELL. D. A. Sillers. *Oil & Gas J.* 26, 25, 146.

When a high pressure gas well is opened into a gas line there is a refrigerating effect, due to the expansion of the gas, which is sometimes sufficient to freeze and shut off the line. It is evidently the water content mixed with the gasoline which freezes, and it is necessary, therefore, either to heat the gas or liquid above freezing point at the place of expansion, or to remove the liquid before the temperature is lowered to freezing. The latter method is generally more desirable.

One method of counteracting freezing is by installing a by-pass around the main valve including a length of small bore tubing, the size and length of this tubing being chosen to carry the full output of gas. There is a heating effect due to friction in this small tube which may counteract the refrigerating effect due to expansion. The tubes are, in effect, choke nipples, and may vary from about $\frac{1}{4}$ in. to $\frac{3}{4}$ in. diameter, and from about 3 ft. to 19 ft. long. The simple by-pass nipple arrangement has worked satisfactorily where pressure drops do not exceed 500 lb./sq. in.

Where very large pressure drops are encountered it has been found effective to drop the pressure through nipples by stages and to install a separating vessel between each stage where condensed liquid may be collected and drained out.

A typical well working on the latter system had a rock pressure of 1,150 lb./sq. in. and produced 51,000 M.C.F. on open flow. It was desired to discharge into a line at 150 lb./sq. in. and several arrangements were tried before a satisfactory one was obtained. The present arrangement consists of a first stage choke nipple $\frac{1}{4}$ in. diameter by 3 ft. long discharging into a vertical drip vessel, a second choke nipple leading from this vessel to a second vessel, the choke also being $\frac{1}{4}$ in. by 3 ft. and a third choke nipple $\frac{3}{4}$ in. diameter by 3 ft. long discharging into the main. The first choke lowered the pressure to 750 lb./sq. in. at 56°F. and deposited about 4 gallons of liquid per hour. The second choke lowered the pressure to 600 lb. at 42°F. and deposited about 10 gallons of liquid per hour, while the third choke lowered the pressure to 170 lb., the line pressure, and discharged 135 M.C.F. per hour at 14°F. The well pressure fell during 24 hours to about 1,075 lb./sq. in., and that and the temperatures and deliveries have remained practically constant. E. R. B. S.

134. INGENIOUS TOOLS FOR FISHING JOBS. T. F. Smiley. *Oil & Gas J.* 26, 25, 86.

Although there is not a tool made for every kind of fishing job which may be encountered, a large number of tools are available which will tackle most problems. It is usual to take a wax impression of the broken end of the tool in the well to ascertain its position and to choose the type of fishing tool best suited for the job, and, if necessary, to design a new type of tool to meet the particular case.

The main features of certain fishing tools are mentioned, such as the Bell Socket Spear, which may be used for removing collapsed or broken casing, and the Eccentric Combination Socket for recovering bits and other tools lost in cavey holes.

Acids are used in certain cases to soften the limestone formation in which casing or tools may be frozen. Experiments are now being made to determine the practicability of dissolving away frozen in casing or tools where other means of removal prove unsuccessful. E. R. B. S.

135. Seminole Air-Gas Lift Installations. Clifton R. Swartz. *Oil and Gas J.*, Vol. 26, 22, 109.

The Seminole Pool at its inception contained sufficient gas to cause natural flow of the oil, but since no free gas was found in the main productive horizon, the Wilcox sand, where all gas present was dissolved in the oil, the natural flowing life was very short, rarely lasting more than 45 days. When natural flow ceased high fluid levels were common, and since efficient pumping was rendered difficult by the depth and crookedness of the holes the gas lift was used almost exclusively for raising oil. Quantity of production was the aim of operators, and wells were originally flowed between tubing and casing, this giving a greater flow than through the usual size of tubing employed: 6¼-inch casing was commonly used with 5 21/32-inch liner set on top of the oil sand; 2 and 2½-inch upset tubing was used to introduce the gas. Tubing depths were frequently changed to increase daily production. When production fell off so that the area between tubing and casing was excessive for the flow obtained, the flow was sometimes changed over from casing to tubing, so producing an economy in gas consumption. Many of these changes proved unfavourable to the operators as production fell off and more than offset the economy in gas requirements. In a few cases tapered tubing was used, usually with successful results, the tubing ranging from 2 in. at the bottom to 5 in. at the top.

Starting pressures varied mainly between 250 and 750 lb. per sq. inch, although higher pressures are recorded, and in one well a pressure of 1,400 lb. per sq. inch failed to start the flow. No attempt was made to reduce starting pressures by rocking or super-flushing. Flowing pressures varied considerably, depending on the method of flow. Tapered tubing required pressures as low as 50 lb. per sq. inch, while flowing through tubing of uniform diameter in some cases required 700 lb. per sq. inch. The majority of wells were flowed between tubing and casing, and had an average working pressure of 190 lb. per sq. inch. The quantity of gas or air required ranged from 400,000 cu. ft. to 1,200,000 cu. ft. per day, the average being from 750,000 to 800,000 cu. ft. In cases where operators continued to flow between tubing and casing after production had fallen off, the gas consumption rose to a high figure per barrel of oil raised. Well-head connections were simple when flowing through the casing. A suitable tubing packer, of sufficient strength to withstand starting pressures, was screwed into the control head or direct on to the casing. A pressure-gauge at the well-head recorded the working pressure. Oil-flow lines were 4-inch and 6-inch, and connected directly to the control head or casing. The general practice was to eliminate all right-angle bends between the well-head and trap, to reduce frictional resistance. Traps were placed close to the derrick and sufficiently high to enable oil to flow by gravity from them to the stock tanks. They varied in size from 4 ft. by 12 ft. to 8 ft. by 20 ft. The first installations did not provide connections for a change over from flow through the casing to flow through the tubing. The compressor plants ranged in size from the small portable plant of three compressors to large semi-permanent or permanent installations of as many as 30 compressors. Compression was always in two stages, and the smaller compressors were belt-driven from 75 or 100-horse power electric motors, while the larger units were direct-driven by gas engines. Compressor size varied from 9½ in. by 4 in. by 10 in. to 16 in. by 8 in. by 20 in. Two general plans of air and gas distribution were used. The header system in connection with a central plant supplying a large number of wells was most common, but in some cases a batch of compressors were connected to an individual well. The majority of plants provided a working pressure header and an extra high pressure starting header. In some cases inter-

mediate headers between high and low compression were provided, which, with fuel and cooling water, made as many as seven headers per plant. Almost all plants allowed for the extraction of gasoline and cooling of the gas between high and low compression, but in many cases this was not repeated after passing through the second compression stage. Approximate overall costs of compressor plants are given and a figure of about 9 cents per 1000 cu. ft. of gas as an approximate working cost.

136. Pipe Line Corrosion is Big Problem. Thornhill. *Oil and Gas J.*, **26**, 31.

Corrosion in pipe lines can be avoided if all moisture is excluded from the pipe surface. Therefore, it is good practice to coat the piping with some material likely to prevent access of water. The ideal permanent coating must be waterproof, unaffected by soil acids and alkalis, have and maintain good adhesion to the pipe surface, must be incapable of adsorption of the surrounding soil and unaffected by ground temperature changes. It must also be a dielectric and capable of being applied in the field without detriment to its properties. A substantial thickness of at least $^1/_{32}$-inch is required, and experiments have proved bituminous or coal tar coatings most suitable.

A. R. S.

137. Drilling for Gas in Appalachian Area. E. W. Williamson. *Oil and Gas J.*, **26**, 34.

This paper describes the cycle of operations which must be undertaken when drilling a gas well. After the site has been chosen and a permanent road has been constructed to carry plant and materials, spudding-in is commenced. Power is obtained from a locomotive type boiler 30–40 h.p. It may be coal or gas-fired ; in the latter case the supply is carried through a 2-in. line. Water supply and a storage tank 250 barrels capacity is also arranged. A 30–40 h.p. steam engine is used.

Spudding-in is continued to a depth of 150 ft., after which drilling is commenced, the depth being increased 5 ft. at a time and the hole being bailed out.

The average well in this field is drilled to about 3,000 ft., and three strings of casing, 10, 8¼ and 6¼ in. are used, and one string of tubing 2–4 in.

A gas well should not be connected to the pipe line without the provision of a drip-line near the well to remove water.

If the production suddenly ceases it may be necessary to bail the well to remove water. If this has entered owing to the casing becoming defective the latter must be pulled and renewed.

P. D.

138. Sounding Apparatus for Determining the Surface and Bottom Levels of a Liquid from a Point above it. L. Steiner and V. Gutman. E.P. 280,809, Nov. 24, 1927.

The sounding device described is more particularly suitable for determining the characteristic of oil soundings. The apparatus consists of a winch and a drum on which is wound a length of two-conductor cable, the cable passing over a wheel and counter, constituting a measuring device, and connected to a plummet which may be lowered down the well for the purpose of sounding.

The winch is arranged to be conveniently and quickly located over, or moved away from, the well, and the cable drum contains a battery and buzzer

connected in series with the fast ends of the two-conductor cable. Slip rings on the shaft of the drum are also connected to the cable ends and means are provided for disconnecting the battery and buzzer.

The plummet may be in several forms, of which some are described. It may contain a mercury switch connected to the two conductors of the cable, the switch normally being open but arranged in such a manner that, upon reaching the liquid surface, a float tilts the switch so as to close the circuit and sound the buzzer on the winch. A feeler on the bottom of the plummet may be arranged so that, upon lowering still further through the liquid until the bottom of the well is reached, the mercury switch is tilted back to its normal position and the circuit again broken, so stopping the buzzer.

In order to ascertain the surface of separation between oil and brine the switch in the plummet may take the form of two electrodes insulated from each other but connected to the cable conductors. The casing of the plummet is arranged in the form of a liquid-containing vessel capable of sliding vertically on the plummet. A small quantity of conducting liquid is placed in the bottom of the casing, but insufficient to reach the electrodes when the casing is in its lower position. On lowering the device to the oil surface in the well the buoyancy of the casing will allow the plummet with its electrodes to descend into the electrolyte, thus completing the circuit and indicating the level of the oil surface. By repeated raising and lowering while immersed in the oil the electrolyte may be expelled through openings in the upper part of the casing, when the circuit will again be broken. As the plummet is still further lowered into the brine, the brine will enter through the holes in the casing and complete the circuit between the electrodes.

The distance of the plummet below ground level is measured continuously by the device on the winch.

By disconnecting the battery and buzzer and connecting suitable apparatus to the slip rings the electrical resistance of liquid in a well may be measured.

In the plummet could also be arranged transmitters of thermometers, pressure gauges, devices for measuring specific gravity or the like, which give their indications at a distance. The cable would then contain a correspondingly larger number of conductors and be connected to an equal number of slip rings on the drum. E. R. B. S.

139. Process and Apparatus for Refining Petroleum and Treating Oil-Field Emulsions. L. E. Winkler and F. C. Koch. U.S.P. 1,650,813, Sept. 20, 1926.

A charging pump forces oil-field emulsions or petroleum through a pipe to the first of a battery of evaporators arranged in stepped relation. Each evaporator has an overflow pipe arranged so that the liquid contents shall maintain a fixed level in each evaporator and shall flow by gravity to the next adjacent evaporator. The last and lowest has a discharge pipe through which the cracked or finished product is conducted to a storage tank.

In order to provide a heating means, highly heated gaseous products of combustion from any suitable source are led through pipes and discharged through a suitable perforated nozzle below the surface of the oil in the first evaporator. The residue gases and vapours pass out of the upper part of the evaporator through a pipe, and are discharged through the nozzle of the next evaporator, this step being continued throughout the battery. The last evaporator discharges to a heat exchanger and from thence the cooled gases pass to a scrubber for removal of the more volatile elements. H. G. S.

See also Abstracts Nos. 1, 2, 6, 8, 74 and 76.

Petroleum Geology.

140. Electric Geophysical Method used in Search for Structure. S. von Croy. *Oil and Gas J.*, **26** 34.

There are four standard geophysical methods in use :—

(1) Measurement of density and its local changes by the torsion balance.
(3) Magnetic measurements (Magnetometer).
(3) Conductivity of artificially produced explosive waves. (Seismograph).
(4) Conductivity of artificially produced electric currents. (Elbof.)

In the latter method the current is sent into the earth by means of point electrodes consisting of metal bars about 1 ft. long, or large metal plates which are buried. Another arrangement is to use two non-insulated cables buried parallel to each other some distance apart. The magnitude of the A.C. generator depends on the depth to which it is desired to investigate.

The strength and direction of the magnetic field in vertical and horizontal planes is then measured by means of a magnet placed at the centre of a frame aerial.

The older Sonde method consists of measuring the current flowing between two searching sondes, and by this means a map can be drawn giving the direction of equipotential lines.

The frame aerial may also be used with a telephonic attachment to indicate when the induced current in it is a maximum.

Another method employs for induced current transmission. Instead of earthed contacts an insulated cable is used. The receiver is similar to the Elbof aerial. The depth of penetration does not exceed 300 metres.

The interpretation of the results obtained by these methods is a matter of practical experience. P. D.

See also Abstract No. 7.

Coal and Shale.

141. Liquid Fuels Other than Petroleum. Paper read before and published by permission of the Institute of Fuel. A. E. Dunstan and H. G. Shatwell.

Despite the continual discoveries of new deposits of petroleum, the rapid increase in production and the recent heavy falls in prices, intensive searches for indigenous liquid fuels in coal-bearing countries have been in progress for some years, whilst important investigations have been conducted in which the conversion of solid into liquid fuels is the chief aim. These activities are influenced to only a small degree by economic considerations, but have their source almost entirely in political and strategic movements. The economic factor involved arises from the fact that solid fuels introduce handling costs of the first importance. For this reason coal might be increasingly gasified at the pit-head and the railroads utilised as means for laying pipe lines to the great towns, the gas being conveyed under high pressure to the existing low-pressure receivers. The carbonisation of coal at low temperatures gives smokeless solid fuel as the main product and primary tar as a by-product, the latter being obtained in yields of about 16 gals. per ton of coal carbonised. The tar-acids present in the tar to the extent of about 33 per cent. cannot be employed as motor-spirit, though they have been used with some success in Diesel engines : their chief potential value lies in the possibility of converting them into benzol hydrocarbons. The tar also contains substantial proportions of low-grade pitch. The oils remaining after removal of phenols

and pitch resemble "cracked" rather than natural petroleum, and their refining must be modified accordingly. The authors have found that primary-tar straight-run spirit possesses an anti-knock equivalent equal to that of benzol, whilst the spirit obtained by cracking the tars also has anti-knock properties. Hydrogenation of coal by the Bergius process gives an average yield of 140 gals. of liquid products per ton of coal. The economics of berginisation will depend largely upon the cost of hydrogen, and it is for this reason that Bergius has recently evolved an ingenious method for recovering hydrogen from the gases leaving the plant. The liquefaction of coal may also be effected by transforming the material, after a preliminary carbonisation, into water-gas, which is treated under different conditions to give a variety of products. Thus the Badische Co. and Patart manufacture synthetic methanol by passing water-gas at high pressure and at about 420° C. over certain catalysts from which iron must be rigorously excluded. Fischer, on the other hand, prepared synthol under similar conditions of pressure and temperature, but by the employment of iron coated with alkali. Both methanol and synthol possess properties which militate against their employment as motor-spirits, but they will readily find application in other directions. Finally, by passing purified water-gas over iron, nickel or cobalt at atmospheric pressure and at temperatures of 150–270° C., Fischer and Tropsch have succeeded in preparing a product which consists entirely of hydrocarbons, and is to all intents and purposes synthetic petroleum.

H. G. S.

142. Process for Elaborating the Products Arising During the Liquefaction of Coal by Hydrogenation. Deutsche Bergin-Akt., fur Kohle in Erdolchemie. E.P. 262,738, Dec. 15, 1927. Appl., Nov. 5, 1926.

Oils are obtained in the hydrogenation of coal, which contain considerable quantities of solid ingredients and by no means inconsiderable amount of asphalt. The product can be worked up by distillation, but it is unavoidable to prevent the production of a quantity of asphalt containing insoluble solid substances.

In this process gas oil from petroleum is added to the product, which has been previously freed from water and low-boiling ingredients, and the mixture of asphalt and solid insoluble substance precipitated is separated from the liquid mixture of coal oil and gas oil. Only a relatively small proportion of gas oil is required thus rendering the process economically feasible, if the work is done at a suitable temperature. The process can be carried out, so that the solid insoluble matter is first precipitated out with a relatively small proportion of asphalt, whilst the major portion of the asphalt is afterwards nearly all precipitated. The temperature conditions required for this fractional precipitation vary with the nature of the oils used.

By this method the distillation of valuable lubricating oils is not disturbed by the presence of easily decomposable asphalt. Further, the gas oil may be so selected that its boiling limits are such that it can be easily recovered by means of distillation.

R. P.

143. Improvements in the Manufacture of Valuable Liquid or Other Hydrocarbons or Derivatives thereof from Carbonaceous Materials. I. G. Farbenindustrie. E.P. 257,912, 1926.

In the hydrogenation of coal tars, mineral oils and the like at high temperatures and pressures, great difficulties arise at times by reason of the high sulphur content of the initial materials, the sulphur generally finding its way into the final products and being difficult of removal.

Desulphurisation is effected by acting on the raw material with water or water vapour, with or without addition of hydrogen, under elevated pressure and at temperatures higher than that at which decomposition of the material takes place. A catalyst is also preferably employed. The sulphur is mainly converted into hydrogen sulphide, which dissolves in the water under the high pressure when the mass is cooled. The pre-treated material is then hydrogenated in the usual way. As an example, a crude mineral oil containing about 1·0 to 1·4 per cent. of sulphur, together with five times the amount of water vapour theoretically required, was passed over a contact mass containing iron and manganese at between 500° and 550° C. under a pressure of 200 atm. The purified oil with a sulphur content of 0·05 to 0·10 per cent., was then hydrogenated at 500° C. and 200 atm. pressure in presence of a catalyst containing molybdenum. Between 60 and 80 per cent. of the oil was converted into benzine. H. G. S.

144. Improved Method of Distilling Oil from Shale. C. A. Spotz. E.P. 281,769, Dec. 9, 1927. Appl., Sept. 9, 1926.

The essential parts of the patent consist in a retort containing liquid metal, an automatic feed and discharge device, and conveyors within the retort to carry the spent shale over the surface of the metal towards the outlet and to convey the raw material beneath the surface of the metal until decomposition and disintegration are complete.

The shale is fed into the apparatus in relatively large pieces and carried beneath the surface of the metal by a combined conveyor agitator and screen. As decomposition proceeds, the pieces of shale gradually become disintegrated into fine particles which pass through the screens and float, to be carried towards the outlet. The vapours rise through the metal, pass out of the apparatus and are condensed. R. P.

See also Abstracts Nos. 13, 32, 33, 72, 86, 98 and 113.

Abstracts.

Arranged by W. H. THOMAS, A.R.S.M.

Abstractors.—S. F. BIRCH, Ph.D., D.I.C., W. S. CLARKE, B.Sc., S. W. COOKE, B.Sc., P. DOCKSEY, B.A., H. S. GARLICK, B.Sc., A.I.C., J. E. MARSHALL-HALL, A.R.S.M., G. SELL, H. G. SHATWELL, Ph.D., M.Sc., A. R. STARK, B.Sc., A.I.C., W. H. THOMAS, A.R.S.M., T. WISKIN.

General.

145. CAN AZNEFT COMPLETE THE PROGRAMME? M. Gallai. *The Oil Bulletin*, 1927, No. 24, p. 4.

The estimated programme for the current operating year forecasts a throughput of 5 mill. tons of petroleum. The refineries of Azneft entered upon 1927–28 operating year with a capacity of only 4·6 mill. tons a year, and to raise this figure to programme demands, it will be necessary to complete and exploitate: (1) The benzine tubular installation on Works No. 5, II. group, by which the yearly productivity of this works will be enhanced 140 thous. tons This should commence operating on Jan. 1, 1928, and therefore will give an increased capacity of 114 thous. tons (2) Two German tubular installations capable of a yearly throughput of 328 thous. tons, but as these will be in operation only from the beginning of the second half of the year the increase will be 164 thous. tons.

With a view to creating some reserve stock on the current operating year, and to prepare the necessary capacity for 1928–29, the following installations were included in the capital expenditure programme.

(1) One tubular battery, "Bakunit" type to distil 278 thous. tons, or near to it, and by this means the total quantity of distilled oils will reach the projected 5 mill. tons.

It would appear therefore that Azneft can fulfil its programme, but if the ancient distilling installations of Azneft with a yearly capacity of 4·3 mill. tons, planned to work 360 days a year, are taken into consideration such a pleasing conclusion cannot be arrived at. To fulfil the programme, with the refineries working at the utmost capacity and nothing in reserve, can only be done during the current year under exceptionally favourable conditions. The least delay, accident, stoppage of the worn-out battery or a greater part of it, to say nothing of the possibility of fire, may for a long period remove out of action a large section of the installation, and substantially decrease the throughput. To fulfil the programme then will be absolutely impossible, with a menace to the plan for supplying oil products to the interior as well as to the exterior markets.

Only with the completion of the 10 in. Baku-Batoum pipe-line, and the conversion of 164 thous. tons of petroleum yearly into benzine, kerosine and gas-oil, can the plan be accomplished.

(2) Two gaso-benzine tubular installations to increase their yearly output by 200 thous. tons.

(3) One plant with a yearly distillating capacity of 820 thous. tons of petroleum for benzine, kerosine and gas-oil.

It was decided to accelerate the erection of Nos. 1 and 2, but under the most favourable conditions these two installations could only commence exploitation during the second half of the year.

Lubricating oils are not taken into account, as Azneft's installations for distilling these are considered to be sufficient to fulfil the programme requirements.

It is considered that the erection of the projected installations during the current year will increase the productivity of the kerosine batteries by 836 thous. tons and by the beginning of the next operating year bring up the total output of the refineries to 5·5 mill. tons. Certain of them will be completed during the current year in time to take part in the work, and distil a minimum of the kerosine and with the two kerosine-oil batteries with an estimated combined distilling capacity of 2·3—2·5 mill. tons it will be possible for Azneft to work more or less calmly, especially so if the projected distilling installations in Baku are completed. At present the position is very acute, and with the least delay in the erection of the works in Batoum and Baku, the difficulties in distilling will be augmented and the disproportion between production and distilling will not only not be overcome, but will unavoidably increase.

T. W.

146. Turbine Drilling and Safeguarding the Interior. General News. *The Oil Bulletin*, 1927, No. 24, p. 10.

Owing to water washing down past the shoe of the pressed-in casing while turbine drilling in Khorosan, the Central Geological Committee discussed the expediency of taking measures to safeguard the petroleum strata from becoming water-logged. The Azerbaidjan Mining Department considers it impossible to prevent water-logging when applying the turbine drilling in its present state and suggest the following measures: (*a*) To strengthen the construction of the turbine drilled wells either by increasing the number of pressed-in columns or by obligatory tamping with cement solution; (*b*) to absolutely prohibit turbine drilling in its present condition on entirely new areas and on the domes of folds; (*c*) to permit turbine drilling on new sections of old areas only after discussion of the projected wells with the C.G.T.C.

The C.G.T.C. does not consider there is sufficient evidence to confirm the water-logging through turbine drilling, and suggested applying this system only on old areas and to the old upper horizons; also to instruct all concerned to pay serious attention to the construction of the well and proposed location for turbine drilling and to the success of the water shut-off.

(Note.—C.G.T.C. = Central Geological Technical Committee.) T. W.

147. Seven Hours Working Day in the Oil Industry. General News. *The Oil Bulletin*, 1927, No. 24, p. 10.

This has now been decided as a practical proposition, and it is proposed to introduce a seven hours' working day first into those branches of the industry where they are connected with the introduction of a second and third change of shifts, as the realisation of this problem in continuous work is more complicated and requires further careful working out.

Much material is now being examined on this subject, and in the near future instructions will be issued for a seven hours' day for those continuously employed (production, drilling, refining). T. W.

148. Azneft-trust. General News. *The Oil Bulletin*, 1927, No. 24, p. 11.

In Batoum at the refineries, 102 reservoirs of various capacity are to be erected for the storage of crude oil and its products. The erection of a number

of huge reservoirs of 6·5 thous. tons each for crude oil has already begun. These are of the latest American type with flat roofs upon which a few inches of water is poured to prevent the escape of volatile products.

A temporary auxiliary electric station of 75 h.p. is now working. Two motors are running and a third 75 h.p. motor is being installed.

The main power station, in which two Diesel engines of 500 h.p. are to be placed is now being erected. T. W.

149. Work of the Neftesyndicate in Batoum. *The Oil Bulletin*, 1927, No. 24, p. 11.

The Batoum branch of the Neftesyndicate has begun to erect 4 benzine reservoirs of 16·4 thous. tons capacity, costing 250 thous. rbls. Four reservoirs of the same capacity for lubricants, costing 240 thous. rbls., will also be erected. The work of changing the old pipe-line on the mole for new 10 in. and 12 in. has commenced, the 12 in. requiring 3500 metres of piping. These pipelines will be served by two lesser steam pumps capable of delivering 260 thous. tons per hour.

Fire precautions are being strengthened. The benzine reservoirs are built in pairs, surrounded by a party-wall 3 m. high. Besides this the undertakings are concentrated in groups which will simplify and cheapen their administration. T. W.

150. Grosny. General News. *The Oil Bulletin*, 1927, No. 24, p. 12.

New Fountain. On November 24, well No. 25/0 in the Soliony Balka, drilled by the rotary system to the XI. horizon came in a fountain. The depth is 1050 metres with a 10 in. finishing column and the daily yield about 220 tons of slightly paraffin oil.

Prospecting on Kertch Peninsular. In well No. 1 at Chongelek, at a depth of 219 m., cavings caused the casings to collapse and the tools to be seized, so that further drilling was impossible. At present the derrick is being transferred to another location 298 m. to the North, where the dip of the strata is more moderate. In the abandoned No. 1 well the dip was about 86°.

In well No. 2 at 373 m. fishing work is going on. A derrick is in course of erection at Aktash. T. W.

151. Chronicle. *The Oil Bulletin*, 1927, No. 24, p. 14.

Renewal of the Naftalan Oil Fields. The Naftalan oil field is situated about 17 km. from Geran Station. This oil contains radio-activity and is applied for healing skin diseases. It has also valuable technical qualities. A splendid sewing machine oil is obtained from it. A high quality black varnish, suitable for isolating electric wire, is obtained from the residue.

As a beginning, 2 of the 8 wells will be renewed and also a small refinery.

Azerbaidjan has already received enquiries from Germany for the Naftalan oil, and it is proposed to export part of the products of this curative oil and to distil part locally for the internal market. T. W.

152. Review of California Oil Industry Developments During the Year 1927. Earl W. Wagy. *Oil Age*, 1928, **25** (3), 25-26, 50.

The average daily shut-in production of crude oil in California increased from 58,000 bbls. in January to 93,000 bbls. in August, 1927, a total of about 25,000,000 bbls. being withheld from the market during the year. It is estimated that since 1922 about 100,000,000 bbls. which would have been produced have been held in the ground.

Stocks of light crude showed a decrease of 10,550,000 bbls. for the year, heavy crude and fuel oil increasing 4,300,000 bbls. Stocks of all products

decreased 3,500,000 bbls. to 137,400,000 bbls. Gasoline stocks increased 1,500,000 bbls.

The demand for all products averaged 687,000 bbls. per day, a gain of 6 per cent. over 1926. Domestic gasoline demands increased by over 13 per cent. to 100,000 bbls. daily, and Pacific export markets consumed about 13,000 bbls. of gasoline daily, about half of which went to Australia and New Zealand.

Californian refineries increased their gasoline output to 167,000 bbls. daily, 21 per cent. over 1926, and about 10,000 bbls. per day of cracked gasoline were produced.

Long Beach.—In October, 1927, four wells were completed at depths of 5600 to 6000 ft. with initial daily productions of 2000 to 4000 bbls., and early in 1928 there were 28 wells producing about 43,000 bbls. daily from this deep horizon.

Rincon.—The discovery well was completed in November, 1927, with an initial production of 1000 bbls. daily from 3220 ft. Four other wells have been completed with productions of 600 to 1000 bbls. daily from about 2600 ft.

Alamitos Heights.—The oil sands are at depths ranging from 4600 to 5500 ft., and the discovery well had a production of 1800 bbls. daily from 4747 ft. There are 118 completed wells.

Ventura Avenue.—This field was extended to the south-west in January and to the east in February, extension wells having daily productions of 2000 to 5000 bbls. daily from 5800 to 6300 ft. Early in 1928 a well was completed at 7015 ft. with a daily production of 2800 bbls. of 30° Bé. oil.

Round Mountain.—The first well was brought in in May, 1927, with an initial production of 350 bbls. of 350° Bé. oil from 2073 ft.

Mt. Poso.—This field was extended 2½ miles south by a well completed in December, 1927, giving 300 bbls. per day from 1781 ft. The potential output is estimated at 4400 bbls. daily from 11 wells.

Potrero.—After numerous unsuccessful wildcats a well was completed in September, 1927, with 200 bbls. from 4717 ft.

Buttonwillow.—In August, 1927, a gas well giving 12 million cu. ft. per day was completed and further work is in hand.

Goleta.—In February, 1927, the discovery well came in at 1331 ft. with 400 bbls. daily of 43° Bé oil. Eight wells have since been completed, the average daily production from December being only 231 bbls. A deep test to 5660 ft. was unsuccessful. G. S.

153. Oil Production Outlook for 1928. *Petr. Times*, 1928, **19** (473), 238.

According to Mr. P. Wagner, of the *National Petroleum News*, producers in California avoided an increase in storage stocks in 1927 by shipping the surplus via the Panama Canal. During 1928 stocks may increase by 15 to 20 million barrels.

The restrictive policy in Venezuela is to be abandoned, and as transport to Atlantic or Gulf ports is as low as 30 cents. per barrel the United States should receive a large part of the crude oil produced, which is estimated at 100 million barrels.

In Oklahoma it is estimated that the production from the Greater Seminole area will be 90 million barrels.

Exports from the United States during 1928 are estimated at 10 per cent. less than 1927, reflecting the entrance of Venezuela and the effect of Russian oil exports.

The potentialities of West Texas in 1928 are great, but prices will be low on account of high sulphur content.

It is fairly obvious that there is little hope for improvement in crude oil prices in the near future. G. S.

154. Oil Pollution of Seas and Harbours—and a Remedy. C. S. Garland. *J. Soc. Chem. Ind.*, 1927, **46** (50), 1161-1171.

The three principal sources of pollution are: (1) discharge of contaminated water; (2) cleaning of tanks at sea; and (3) accidents.

Unlike other sources of pollution, the contamination of the sea by oil is persistent and cumulative, and this has been proved by experiment.

In both England and the United States it is an offence for oil to be pumped overboard in territorial waters.

The effect of pumping oil overboard is: (1) loss of oil; (2) danger of fire in harbours, etc.; (3) damage to paint work of other vessels; (4) destruction of fishing nets, driving away of fish and destruction of immature fish; (5) discomfort to persons using beaches, and (6) destruction of sea birds. While not of economical importance, the last is the greatest reflection upon civilisation, as the birds die a lingering and painful death.

The loss to seaside towns is important, and in one case out of 100 possible bathing days the presence of oil prevented bathing on 23 days.

The economic loss is far more serious, and it is estimated that the total loss of oil into the sea in one year amounts to 500,000 barrels, valued at £250,000.

There has been some difference of opinion as to whether the oil had any deleterious effect on fish, but this seems to have been settled by Mr. D. Hayden Roberts from experiments published in 1926. Three-inch fish were immersed in sea water in which oils varying from thin Diesel to heavy asphatic crude had been shaken up. The fish rapidly lost equilibrium and died in 3 to 26 minutes.

Large oil-using ships pump from 1 to 2 thousand tons of ballast water after each voyage. The average oil content is from 2 to 3 per cent., about 95 per cent. of which floats on top of the water and will be discharged in a body.

It is important that the time lost in port should be short and, therefore, pumping operations should not occupy more than 10 hours. The oil-water mixture must be dealt with at the rate of 100 to 200 tons per hour, and the effluent water must not contain sufficient oil to form a film on the water. Calculations show that 1 gallon of fuel oil will produce a film over an area of more than 1100 acres. The Admiralty specification for a satisfactory plant demands a test of 200 tons per hour for 10 hours, the oil content of the ballast water varying from 5 to 15 per cent. of Navy fuel oil, the average oil content of the effluent water not to exceed 0·0005 per cent.

The first patent for an oil separator was taken out in 1892 and, in common with several others, depended on the tendency of oil to rise through the water and up a tapering channel or the sides of a cone to the oil outlet.

In another type the mixture passes along the bottom of a rectangular tank having an inclined roof, the oil being collected by baffles and ascending, passes in the opposite direction to the oil outlet.

In all the designs, increase in capacity can only be obtained by increase in size. One essential of a satisfactory separator is the absence of eddy currents and applying the criterion of Reynolds the diameter of a simple, circular pipe for a throughput of 200 tons per hour would be 100 ft.

The Hele-Shaw and Beale separator avoids this by the multiplication of the standard tube unit. In its simplest form, the separator consists of a cylindrical vessel containing a large number of 1½ in. diameter tubes through which the mixture flows. The tubes are perforated along their upper surface to provide for egress of the oil into still water.

It is impossible, without a filter or coagulating device, to remove the last traces of emulsified oil, and various means have been suggested and have met with some success on small scale tests.

In the Stream-Line oil separator, glass silk, the filaments having a diameter of 0·009 in., is pressed into an annular basket of galvanised wire mesh or galvanised expanded iron. Glass silk has a surface area of over 9000 sq. ft. per lb., it will attract 2·83 lb. of oil before being clogged, its back pressure is negligible, and it is not injured by steam.

A cure for oil pollution is now available in a separator which can be put in the bilge line and will give an effluent containing less than 1 part of oil in 200,000 of water. Tests were made in June, 1927, on an Admiralty barge fitted with this separator and two lots of 1000 tons of water pumped through in 10 hours. During the first period the mixture contained 1 to 2 per cent. of oil in tank washings, and during the second 5, 10 and 15 per cent. of Navy fuel oil in sea water. Owing to the viscous nature of the oil it was necessary to run the plant at over 300 tons per hour at the end of each tank to maintain the average.

The cost of fitting separators is given as an objection against compulsory fitting. An oil-burning vessel, when in ballast, carries 600 to 1500 tons of water containing at least 1 per cent. of oil. A plant of 100 tons per hour capacity costs under £1,000 to instal and, as no additional pump is necessary, would earn a return of at least £30 per voyage.

The examination of samples of effluent containing 0·0001 to 0·0005 per cent. of oil presents difficulty. Three recognised methods of analysis were studied using a standard solution of fuel oil in ether, the ether being allowed to evaporate after shaking with sea water. The methods used were :—

1. Extraction with pure benzine, filtration of the benzine after separation with a separating funnel, and evaporation at low temperature with drying to constant weight at 100° C.

2. A similar extraction with chloroform and drying to constant weight at 100° after filtration.

3. Mixing with a solution containing 0·5 gr. of aluminium sulphate, precipitation with excess of ammonia, filtration of the alumina, drying at 100°, and Soxhlet extraction of the dried precipitate, evaporation of the benzine from the extract at low temperature and drying of the oil residue to constant weight.

Contrary to expectation, nearly all the results were higher than standard, while if filtration was omitted the results were 30 to 70 per cent. high, probably on account of solid particles taken up by the oil. G. S.

155. Process for the Activation of Silicic Acid Gel. I. G. Farbenindustrie Aktiengesellschaft. E. P. 263483. Jan. 16, 1928.

The invention consists of a process for the activation of a silicic acid gel. The gel is first dried until consolidation of the gel structure has taken place, and is then roasted in a revolving furnace at temperatures between 300° C. and 600° C. to obtain a solid product. W. H. T.

156. Emulsifying Agents. I. G. Farbenindustrie Akt. E.P. 268, 387. March 28, 1927. Conv. Sept. 18, 1925.

An improvement is described of E.P. 258,551, which consists in the employment of organic sulphuric acids or salts thereof in conjunction with alkylated cellulose, for the preparation of emulsifying agents and emulsions. These are especially suitable for softening and oiling preparations for use in spinning textile fibres. Particularly good results are obtained when employing such aromatic or hydroaromatic sulphuric acids as contain alkyl-, aryl-, aralkyl- or cycloalkyl radicles, either alone or mixed with one another or salts of such acids. An example given consists of dissolving 2·5 parts of dimethyl cellulose and 2·5 parts of sodium isopropylnophthalene-sulphonate in about 300 parts of water, and adding 80 parts of olein with continuous stirring until a homo-

geneous emulsion is obtained. Instead of isopropylnaphthalene-sulphonate the butyl- or benzyl salts or sulphonic acids may be used, and in place of the olein oilve oil, mineral oils, or mixtures of these oils may be employed.

H. S. G.

See also Abstracts Nos. 176, 204 and 205.

Chemistry of Petroleum.

157. The Partial Vapour Pressures of the Benzene-Toluene and Benzene Ethyl-Benzene Mixtures. T. Bell and R. Wright. *J. Phys. Chem*, 1928, xxxi., 12.

The chief difficulty encountered in carrying out this work was the analysis of the gaseous mixtures. Measuring the density of the vapour gave very inaccurate results owing to the small difference in the densities of the two constituent gases. The analysis was carried out by measuring the depression of the freezing-point of the condensed vapour after it had been mixed with a known quantity of pure benzene.

Determinations for F.P. depression for small additions of toluene and ethyl benzene were made and the results are given. In carrying out the vapour pressure determination the dynamical method was used, the saturated mixture of air and vapour being passed through a weighed liquid air trap to condense the benzene and toluene. The collected liquid was then analysed as described above. The partial pressures are in proportion to the molecular concentration of the liquid; the results indicate that if complex formation takes place in the mixtures considered it must do so only to a very slight extent.

P. D.

158. A Method for Comparing the Time of Mixing of Two Transparent Liquids in Different Proportions, and some Experimental Results, particularly with Gasoline and Carbon Bisulphide. L. E. Dodd. *J. Phys. Chem.*, 1928, xxxi., 12.

The purpose of the article is to describe the apparatus and the suitability of the method used for carrying out the tests. The two liquids used for the tests were commercial gasoline and carbon bisulphide, except in one set of experiments when water and concentrated brine were used. The behaviour on mixing was observed by watching the image formed by a beam of light passing through the liquid. The light field maintains a disturbed and striated appearance which disappears suddenly. The moment at which the field clears is said to be that at which mixing is complete. The precision of the method is raised by the use of a long focus lens between liquid and observation screen. The rate of mixing is affected by numerous factors such as the size and shape of the tank, size, shape and motion of the stirrer. The tank was of rectangular cross-section and held about 60 c.c. of the mixed liquids. The sides were ground flat outside, but owing to the small difference between the refractive indices of the glass and liquid it was not considered necessary to pay particular attention to the inside surface. The stirrer consisted of a rectangular piece of galvanised wire cloth made to move up and down in the liquid in a definite manner. The stirring motion was arranged so that the stirrer rested for part of its stroke on the bottom, and was lifted clear of the liquid for an equal length of time at the top of the stroke. One liquid was placed in the tank, and the other gently run in, care being taken to give as little initial stir and consequent unknown mixing of the liquids as

possible. The stirring was then started; and the time taken for complete mixing was that from the commencement of stirring until the image cleared.

Various methods of introducing the second liquid were tried; in the first runs the liquid was gently poured down the side of the vessel. The method adopted for the later runs was to run the second liquid down a tube which went to the bottom of the tank. The end of the tube was narrowed down to prevent too great velocity of liquid issuing from it. This method was adopted whether the heavy or light liquid was first placed in the tank.

Using the two liquids mentioned above the time of mixing varied from 18 seconds to 6·5 minutes, depending on the composition of the mixture. The results showed a fair degree of reproducibility. Several interesting conclusions are drawn as to the mechanical processes taking place during mixing.

P. D.

159. The Action of Sodium-Potassium Alloy on Petroleum. J. B. Conant and A. H. Blatt. *J. Am. C.S.*, 1927, **50**, 542–550.

The authors found that when a sample of Pennsylvanian crude diluted with dry ether is shaken with sodium-potassium alloy in an inert atmosphere an intense brown solution and precipitate are formed. From the fact that this colour was discharged by introducing moisture or dry carbon dioxide, and that a considerable amount of the latter was readily absorbed forming a white precipitate, the authors inferred that a sodium, or possibly potassium, compound of one of the constituents of the petroleum had been formed. On testing, it was found that this peculiar property was not confined to Pennsylvanian crude but that a number of crude oils, including Mid-Continent, Californian and Colombian, as well as a sample of "cracking-coil residue" from a Mid-Continent gas oil, behaved in a similar manner. No action, however, was observed when "cracking-coil" distillate from the Mid-Continent gas oil was examined.

The reaction itself takes from 12–24 hours of vigorous shaking for completion. After this time, shaking the oil with fresh alloy produces no further action. It is hoped in a future investigation to regenerate the hydrocarbon from the brown precipitate for examination. The white precipitate formed by the action of carbon-dioxide has been shown to be the sodium, or potassium, salt of the carboxylic acid derived from this hydrocarbon. That it was not derived from naphthenic acids present in the crude was proved by carrying out a blank experiment; it was then found that naphthenic acids present only accounted for 11 per cent. of the precipitated sodium salts from the Mid-Continental and only a minute fraction of those obtained from the Pennsylvanian and Colombian oils.

The crude acids, prepared from the sodium salts by acidification, are brown amorphous solids without any definite melting-points; they are probably mixtures. So far attempts to purify them have failed. Analysis of the acids from the Pennsylvanian and Colombian oils indicate that they contain one carboxyl group for each benzene ring present, and that they are free from halogens, sulphur and nitrogen. Attempts to prepare amides through the chlorides by the action of ammonia indicate that the acids are dibasic. Pennsylvanian oil, which had been treated with the alloy at room temperature until no further action occurred, reacted with it at 250° in the presence of carbon-dioxide to give alkali salts. It is possible that under these conditions dehydrogenation may have taken place prior to the reaction with the alloy. The formation of the metallic compounds may be due either to the cleavage of a highly substituted ethane or to the direct addition of the metal to an unsaturated system. The results obtained point to the latter alternative.

S. F. B.

160. The Action of Sodium Potassium Alloy on Certain Hydrocarbons. J. B. Conant and A. H. Blatt. *J. Am. C.S.*, 1927, **50**, 551–558;

The authors have found that sodium-potassium alloy reacts with various samples of petroleum to form highly coloured organo-metallic compounds. (*See* previous Abstract.) In order to investigate the reaction they have prepared a number of pure hydrocarbons and examined their behaviour when treated with the alloy. As the analyses of the compounds obtained from petroleum indicated compounds having one carboxyl group for each aryl group present, the investigation has been carried out on two classes of hydrocarbons which fulfil the necessary conditions. The first class consisted of substituted ethanes and included diphenyldimethyl-ethane, diphenyldibutyl-ethane, diphenyldi-*iso*-propyl-ethane and diphenyl-di-*tert.*-butyl ethane; they were found to be without action on the alloy. The second class consisted of substituted ethylenes such as propylstyrene and dimethylstyrene. Both of these hydrocarbons reacted, and by the action of carbon-dioxide yielded adipic acid derivatives. The action of water on the sodium compound from dimethylstyrene yielded dibenzyltetramethylethane. S. F. B.

161. Some Constituents of Petroleum from Maki, Eeligo Province. Satoyasu Timoni and Uchu Kikuchi. J.C.S., Japan, 1927, **48**, 520–526.

The bromination, nitration and chlorination of fully and incompletely refined fractions boiling between 125° and 155° from Maki petroleum, have led to the separation and identification of derivatives of xylene and of hexahydro-*ψ*-cumene. The partly refined fractions, in the presence of reduced iron or aluminium powder gave tribrom-*ψ*-cumene m.p. 235° C., tetrabromxylene, m.p. 250°, and trinitroxylene m.p. 174°, whilst the partly refined fractions gave tribrom-*ψ*-cumene and trichlor-*ψ*-cumene, thus confirming the presence of the hydrogenated parent hydrocarbon. The fact that the melting-point of the tribrom-*ψ*-cumene did not coincide with that of a genuine specimen was probably due to the presence of hemimellithol. S. F. B.

162. Hydrogen-ethylene Reaction in the Presence of Excited Mercury Atoms. A. R. Olson and C. H. Meyers. *J. Am. C.S.*, 1927, **49**, 3131–3134.

In the presence of excited mercury atoms hydrogen and ethylene react to give various products, among which methane, ethane, propane and butane were detected by means of positive ray analysis. Suggested mechanisms for the reaction are given. S. F. B.

163. Formation of Anthraquinone by Vapour-phase of Cracking Toluene and Toluene Containing Petroleum Distillates. A. R. Bowen and A. W. Nash. *Nature*, 1927, **120**, 621.

By the passage of toluene or petroleum fractions containing toluene mixed with air over aluminium grains covered with vanadium oxide at 400°, traces of anthraquinone, benzoic acid and benzaldehyde were found. S. F. B.

164. Cleavage of the Heterocyclic Compounds of Petroleum. R. Weissgerber and C. Seidler. *Ber.*, 1927, **60**, 2088–2090.

For technical reasons it is often necessary to remove the five-membered heterocyclic compounds which usually accompany the hydrocarbons. Although these may only be present in relatively small amounts, during fractionation they may collect in one fraction, with the result that on refining the loss may be as high as 30 per cent. The usual method of refining is to treat the oil with sulphuric acid, which results in these substances being converted into practically useless resins. The present authors have found that by the action

of alkalies at high temperatures, heterocyclic rings attached to a phenyl group are ruptured with the formation of ortho-substituted toluenes. This reaction, which is partly hydrolytic and partly oxidative, can only be explained on the assumption that formic acid is formed, which, however, may become converted into carbon-dioxide under the conditions employed. Experimentally it was found that when thionaphthene was heated from 300–310° for 4 hours with potassium hydroxide, 74 per cent. was recovered unchanged, but that 83 per cent. of the fission product consisted of *o*-thiocresol. Under similar conditions coumarone and indole gave excellent yields of *o*-cresol and *o*-toluidine respectively. On the other hand, indene yielded over 50 per cent. of a black amorphous mass together with traces of *o*-xylene. Under the experimental conditions diphenylene sulphide, carbazole and thiophene remained unattacked. S. F. B.

165. Cracking of Hexadecane in the Presence of Catalysts. Henry Gault and René Sigwalt. *Ann. Office Nat. Comb. Liquides*, 1927, 2, 309–323; 543–584.

A continuation of work previously described. Using the same apparatus as that described in the earlier Papers, the authors have cracked hexadecane in the gaseous phase at different temperatures, employing an empty quartz tube and also the same tube packed with catalysts. These included grains of quartz, pumice coated with carbon or with reduced nickel. A number of experiments on liquid phase cracking were also carried out, using aluminium trichloride as the catalyst. The gaseous and liquid products were analysed by the method employed in the earlier part of the investigation. In order to obtain comparable results, especially when using catalysts, it was found that it is essential to crack the same volume of hexadecane in each experiment. The results obtained indicated that the cracking reactions take place irregularly, and particularly when catalysts are employed, that dissociation centres are formed. Cracking temperatures can be considerably lowered by introducing a catalyst such as quartz or pumice, but the final products differ from those obtained with an empty quartz tube. The light products, obtained with the empty tube or pumice, undergo considerable polymerisation and in time resinify. Aromatic hydrocarbons are produced from the lower ethylenes, which are the primary products of the degradation, and not from acetylene, which was not present in the gaseous products. In the absence of chemical catalysts the carbon chain appears to break indifferently at any point, but as the temperature increases the fission tends to approach the terminal carbons; the double bond is found in the shorter of the two resulting molecules. There is very little difference in the composition of the gaseous products, which vary within very narrow limits with the temperature. The liquid products are much less stable than the lower gaseous homologues. In the absence of chemical catalysts, such as nickel, which bring about deep-seated decomposition, differences in the composition of the liquid hydrocarbons are due to secondary reactions, for the primary products at various temperatures appear to be essentially the same. The carbon coated pumice was less active at 400° and 450° than pumice alone; this inhibiting effect was less noticeable at higher temperatures. The gaseous products formed in the presence of nickel consisted almost entirely of hydrogen and methane, while the liquid products were few in number and consisted of aromatic and naphthenic hydrocarbons; ethylene hydrocarbons were only found at temperatures as low as 350° C. The activity of the reduced nickel increases to a maximum and finally decreases. The liquid phase cracking of the hexadecane in the presence of aluminium chloride gave chiefly butane and a little propane with small amounts of other substances. An asphaltic residue, poor in hydrogen, was left. S. F. B.

166. THE "UNSATURATED HYDROCARBONS" IN THE GASES FROM THE CARBONISATION OF COAL. A. B. Manning, J. G. King and F. S. Sinnatt. *Fuel Research Report*, No. 19.

The usual methods of gas analysis only allow of the determination of the total percentage of the unsaturated constituents, including aromatic hydrocarbons, if present, and leave undecided the relative proportions of the individual hydrocarbons. A more exact knowledge of the relative proportions of the various constituents is desirable, not only to throw light on the process of carbonisation but also in view of the commercial utilisation of the unsaturated hydrocarbons. Thus a considerable amount of attention has recently been given to the conversion of the gaseous olefines into the corresponding alcohols and glycols, although so far no use has been found for the diolefines. The possibility of converting the latter into synthetic rubber, however, renders the determination and separation of these hydrocarbons of great interest. The methods previously used to determine the unsaturated hydrocarbons consisted in converting them into their bromine addition compounds and subsequently separating the latter by fractional distillation, and in this connection may be mentioned the classical work of Armstrong and Miller, who isolated ethylene, propylene and butadiene from compressed oil gas. The same method has also been employed by a number of other workers, including the present authors, who used it to determine the composition of unsaturated hydrocarbons in coal gas. The method does not, however, lend itself to the quantitative determination of olefines, because as sulphuric acid of varying concentration has also been used to effect fractional solution of the unsaturated hydrocarbons, and although this method may give excellent results with rich gases, it can hardly be applied to "lean" gases such as coal gas. The authors have therefore devised a method involving the separation of the unsaturated hydrocarbons as their bromine addition compounds and the regeneration of the olefines from the latter by means of the zinc-copper couple. The resulting gas is then analysed by (*a*) absorption of propylene and butylene by 87 per cent. sulphuric acid, and (*b*) combustion of a further quantity of the gas over copper oxide at 700° C. and determination of the volume of carbon-dioxide produced.

The apparatus employed for the preparation of the bromides consists of a meter for measuring the gas, followed by a 3-ft. glass tube packed with Lessing rings down which gas oil is allowed to trickle. The gas to be analysed flows counter-current to the oil; about 1½ litres of gas oil are required to scrub 10 cubic feet of gas. Any unsaturated gases which dissolve are recovered later by distilling the gas oil and converting to bromides; these are then added to the main bulk. The gas passed from the scrubber through the Woolf bottles containing bromine covered with a layer of 10 per cent. potassium bromide. Following the Woolf bottles the gases now mixed with bromine vapour pass into the cooled special condensers where the bromine is condensed. This bromine is returned to the second Woolf bottle from time to time. The gases are passed through this apparatus at the rate of two cubic feet per hour. When the absorption is complete, the bromine compounds are freed from bromine by dilute caustic soda, washed, separated and weighed. The bromides, from the olefines recovered by distilling the gas oil to its initial boiling-point, are also added. The mixed bromides are then separated by fractionation at 20 m.m. pressure into three fractions: (*a*) up to 80° C., (*b*) 80°–130° C., and (*c*) residue. Fraction (*a* contains all the ethylene and propylene and most of the butylene as dibromides. The second fraction (*b*) contains some butylene bromides together with any benzene or toluene which may be present, while fraction (*c*) contains acetylene and butadiene tetrabromides. The object of taking the second

fraction is to insure that fraction (*a*) contains no appreciable quantities of homologous bromides higher than butylene dibromide.

The apparatus employed for the regeneration of the olefines consists of a small tube, about 30 c.c. in capacity, fitted with a cork carrying a glass tube connected by a rubber tube to a funnel, which can be raised or lowered, and a small water condenser, the top of which is attached to an ordinary gas burette.

About 2 gms. of fraction (*a*) are weighed into the small tube, which is two-thirds filled with freshly prepared zinc-copper couple covered with 95 per cent. alcohol. The reaction is started by warming. The gas burette is filled with a saturated solution of calcium or magnesium chloride, which serves to dissolve any alcohol which may pass through the condenser. The gases are then transferred to a Bone and Wheeler apparatus and analysed for (*a*) total olefines by absorption in fuming acid or bromine water, (*b*) for carbon by combustion of a portion of the gas over copper oxide at 700° C., and (*c*) for propylene and butylene by shaking with 87 per cent. sulphuric acid.

The results obtained give the necessary data for determining the ethylene : propylene : butylene ratio in fraction (*a*). Fraction (*b*) is treated similarly, and the value obtained for the butylene added to the results from fraction (*a*). Fraction (*c*) is analysed for butadiene by extraction with hot alcohol, which dissolves the high melting butadiene tetrabromide, and cooling to 0° C. The solid tetrabromide is rapidly filtered, dried and weighed. This weight represents only half of the total butadiene present, the other half being present as the alcohol-soluble low-melting form. The results obtained by analysing synthetic mixtures of gases using this method appear to agree well with the theoretical figures; the method has therefore been used for analysing the gases produced by the low-temperature carbonisation of Dalton Main Coal at temperatures between 400° and 700° C. in the plant at the Fuel Research Station. The results in general show an increase in quantity of all olefine hydrocarbons with rising temperatures; the proportions of the three olefines in the gas varies considerably with temperature while the propylene and butylene contents fall. The butylene content falls very rapidly above 600° C.

Butadiene was only found in the gases produced at and above 600° C. Compared with high-temperature carbonisation gas, the gas is much poorer in ethylene and richer in propylene and butylene, due, no doubt, to the severe cracking which occurs in the high-temperature process. S. F. B.

167. Improvements in the Manufacture and Production of Unsaturated Hydrocarbons. I. G. Farbenind Akt.-Ges. E.P. 264,827, 1928.

The production of unsaturated hydrocarbons—*e.g.*, ethylene and acetylene—is effected by passing aliphatic hydrocarbons, *e.g.* methane, over catalytic agents at temperatures of 700° to 1000° C., and pressures equal to or exceeding atmospheric. The catalysts specified are salts of the alkaline earth metals, or salts, oxides or hydroxides of magnesium or beryllium, or compounds of selenium, tellurium or thallium, or active silica or charcoal, or mixtures of the above with each other. The products should be removed as quickly as possible from the hot reaction zone. H. G. S.

See also Abstract No. 175.

Crude Oil.

See Abstracts Nos. 159 and 161.

Natural Gas.

168. Air Filters in Natural Gasoline Plants. O. F. Kluenter. *The Refiner*, 1928, 7, 1, 58.

Unfiltered air is responsible for frequent valve cleaning; replacement and grinding of valves; reboring of cylinders; loss of capacity; excessive consumption of lubricating oil, and carbon deposits and other troubles with compressors. The foreign substance which does the most harm are particles of sand which are of the same thickness as the filament of oil between the cylinder rings and the walls of the cylinder. This causes equipment to pump oil which in turn assists in forming carbon in the cylinder heads. The air filter cannot be too strongly recommended. Properly used, it can clean the air of from 90 to 95 per cent. of the foreign substances, but if the proper efficiency and full benefits are to be derived, the filters must be cleaned at given periods and recharged. The time between these cleanings and re-chargings depends entirely upon the local conditions around the gasoline plant. S. W. C.

See also Abstract No. 181.

Motor Spirit.

169. Making Gasoline from Refinery Vapours. W. G. Palmer. *Oil and Gas J.*, 1928, 26, 33, 148.

Generally, vapour recovery systems are installed to handle vapours from the following sources: (1) Refinery storage tanks; (2) run or receiving tanks directly associated with refining operations; (3) refinery condenser boxes and vents.

The vapour gathering system is the vital part of a recovery installation. To ensure economic success a preliminary survey should be made to ascertain the possible production. In a general way the refining loss indicates the potential production from the recoverable vapours. The initial investment is not the primary consideration in selecting equipment for a recovery plant. The methods of operation of such a plant are: (1) as natural gasoline plant; (2) as an extraction unit; (3) as a blending plant in which gasoline of a high initial B.P. is used as an absorbent medium, through which the still vapours are passed after cooling. The product of refinery vapour-recovery plants is comparatively stable. Rarely is the recovery below 90 per cent. The gravity will vary between 64° and 80° A.P.I. Distillation curves will show the initial boiling points to vary between 85° and 100° F., and will follow the contour of curves from distillations of straight-run domestic gasoline. The end-point, being a factor of individual plant control, is optional. Tabulated data from operating plants are given as examples. S. W. C.

170. A New Method of Refining Pressure Distillates. *Petr. Times*, 1928, **19** (No. 476), 346.

Sulphuric acid is easily the commonest reagent employed in the refining of pressure distillates, and in spite of numerous other reagents it appears to maintain this position.

There can be little doubt as to its efficacy in refining cracked distillates, but its use is attended by many disadvantages. It dissolves and removes from the pressure distillate other substances besides those which it is intended to destroy, some of which can be advantageously retained in the oil on account of their anti-knock value. There is also the problem of acid sludge disposal, which is of considerable importance and expense whether the acid

is recovered or transported to a dump. Sulphuric acid has, however, many advantages to balance these disadvantages.

Among the more recent methods described for refining pressure distillates, that now employed by the Houston Oil Company of Texas is of considerable interest on account of the reagent used, an ammoniacal solution of cupric hydroxide. The oil to be treated is washed free from hydrogen sulphide either by exhaustive water washing or by a small soda wash. The latter takes considerably less time than water alone but is more expensive. The washed distillate is then treated with a moderate amount of sulphuric acid, in the cold, to polymerise diolefines. After sludging, the new reagent is employed. This is prepared by adding sufficient caustic soda to a solution of copper sulphate to precipitate all the copper as hydroxide. Sufficient ammonia is then added to dissolve the precipitated hydroxide; the solution is then ready for use. The distillate is agitated with this solution in small amounts; the blue colour is at first destroyed and fresh solution must be added. When the colour is no longer discharged the gasoline is removed and steam distilled. The distillate is stated to conform to the most rigid specifications. The explanation offered is that copper is capable of forming compounds with acetylenes, diolefines and certain sulphur compounds, such as mercaptans and disulphides, which are insoluble and, it is stated, separate as a green precipitate. Silver acts in the same way, but is ruled out on account of its high cost. Steps are being taken to protect the process.

S. F. B.

171. Improvements in the Purification of Crude Benzol. I. G. Farbenind Akt.-Ges. E.P. 258,576, 1926.

Crude benzol is treated at a high temperature and under elevated pressure with hydrogen or gases containing or giving rise to hydrogen in the presence of catalysts such as contact masses containing chromium, tungsten, molybdenum and the like. The product, when distilled, yields pure benzol. Thus a crude benzol recovered from a saturated wash oil, when treated with a mixture of hydrogen and nitrogen at 450° C., and under a pressure of 150 to 200 atm. in contact with a pressed mixture of molybdic acid and magnesium oxide gives pure benzol and a mixture of other valuable hydrocarbons of low boiling point. Similarly, a crude benzol containing thiophene can be obtained in a pure state by interaction with hydrogen in an aluminium-lined autoclave at 460° C. and 200 atm. pressure, the contact mass being molybdic acid and zinc oxide.

H. G. S.

172. Improvements in the Manufacture and Production of Motor Fuels. I. G. Farbenind Akt.-Ges. E.P. 259,944, 1926.

The motor fuels specified are prepared by blending a usual motor fuel, especially one which is inclined "to knock" in the engine, with a product substantially composed of liquid hydrocarbons obtained by the destructive hydrogenation of coals, tars, mineral oils, distillation and extraction products and residues thereof or any other carbonaceous material. For example, a benzine of 0·730 specific gravity, consisting chiefly of aliphatic hydrocarbons, is mixed with 40 per cent. of a product boiling below 200° C. produced by the destructive hydrogenation of brown coal. The resulting mixture allows a motor to be operated with a high compression ratio—*e.g.*, 5 : 1.

H. G. S.

173. Purification of Benzol, Petrol and the Like. K. Cox and P. J. McDermott. U.S.P. 1,658,285. Feb. 7, 1928.

The invention comprises the treatment of spirit with ferric sulphate or persulphate of iron, $Fe_2(SO_4)_3$, during distillation. A microporous material

can be added to the iron salt in order to assist in the polymerisation of undesirable unsaturated hydrocarbons. The distillate is afterwards washed with a weak alkaline solution or is filtered through an alkaline material such as lime. W. H. T.

174. Removal of Sulphur and Sulphur Compounds from Hydrocarbon Oil. G. L. Wendt. U.S.P. 1,658,505. Feb. 7, 1928.

The patent describes a process whereby sulphur is removed from gasoline, kerosine, etc., by washing the oil with an alcoholic solution of sodium plumbite. The strength of alcohol should be at least 50 per cent., and the treatment can be preceded by a wash with aqueous sodium plumbite solution.
W. H. T.

See also Abstracts Nos. 157, 158, 181, 185, 200, 209, 214, 231, 248 and 249.

Kerosine.

175. On the Desulphurising Effect of Silica Gel and on the Failure of Lamp-burning in the Presence of Mercaptans. By H. I. Waterman and M. J. van Tussenbrock. *Brennstoff-Chem.*, Vol. IX., No. 3, p. 37.

In these experiments aliphatic sulphur compounds in solutions of kerosine were utilised. A distillate of a British Indian petroleum served as kerosine whose sulphur-content by the lamp method was 0·018 per cent. In one case with dipropylsulphide a commercial kerosine with a S content of 0·078 per cent. served as the solvent. The sulphur compounds in question, with the exception of diethylsulphide and ethyl mercaptan, were prepared by the Eastman Kodak Co., Rochester, N.Y. These were submitted to distillation tests before experiment and exhibited the following characteristics :—

1. Dipropylsulphide $(C_3H_7)_2$ S. B.Pt. 140·5-142·5° (141·5—142·5° in the literature).
2. Diethyldisulphide $(C_2H_5)_2$ S_2. A distillate from the original product boiling between 153·0—153·9° was used (B.Pt. 152·8—153·4° C. in the literature).

The refractive indices of these preparations are also given :—

3. Dipropyldisulphide $(C_3H_7)_2$ S_2. B.Pt. 192·5° (192·5° in the literature).
4. Ethylmercaptan C_2H_5SH. The fraction boiling between 34·1—37·0° in the redistillation was utilised.
5. Isopropylmercaptan $(CH_3)_2CHSH$. 7·51 gms. were distilled. The product commenced to boil at 49·4°, 1·45 gms. distilling over up to 52·4°. The principle quantity of 5·97 gms. boiled between 52·6—55·0° and a small quantity of a bright yellow residue remained. (B.Pt. 57–60·0° in the literature, B.Pt. 53–66° Eastman Kodak Co.).
6. *n*-Butylmercaptan C_4H_9SH. The principle quantity boiled between 96–98°, and a small residue remained. (B.Pt. 97–98° in the literature, 96–98° Eastman Kodak Co.)
7. Isoamylmercaptan $(CH_3)_2$ $CH(CH_3)_2SH$.

Gms. substance used.		Boiling limits.		Gms. distillate.		B.Pt. (in the literature).
22·66	..	99–116°	..	0·36	..	—
—	..	116–118°	..	12·92	..	116·6–118°
—	..	118–120·5°	..	8·59	..	—
—	..	Residue	..	0·42	..	—

Solutions of the sulphur, disulphide and mercaptan in kerosine were prepared, whose known S contents were again tested by the lamp-method in

the presence of silica gel. In the case of diethylsulphide about one-third of the sulphur was adsorbed by the silica gel. No special difference was observed in the desulphurising influence of both dialkylsulphides. It is suggested that this may be due to the decrease in the adsorptive capacity of disulphides with increasing molecular weight. The size of the grain of the silica gel appears to have no effect.

The lamp-method fails for solutions containing mercaptans, and this has been shown not to be due to deterioration of the sulphur compounds used. The sulphur contents of *n*-butyl mercaptan and isoamylmercaptan were also determined by the Carius method. The following table gives a comparison of the results obtained :—

	C_4H_9SH	$(CH_3)_2:CH:(CH_2)_2SH$
Calculated S content of kerosene solution	1·018	3·030
S content obtained by Carius method	0·975	3·102
S content obtained by combustion method	0·851	2·744

An analysis of the isoamylmercaptan by the Carius method gave the value 30·74% Sulphur, the calculated value being 30·77% S.

It was found that in the burning of mercaptan solutions in the lamp considerable quantities of sulphur were deposited on the wick. Glass capillaries, instead of wicks, were utilised in this research, and the lamp was further provided with a "washer" fitted with a cock so as to enable a "rinsing" with benzene or alcohol. In this way a complete combustion of the original solution was ensured. However, the following points had to be established by repeated experiments before the new modification of the lamp could be used : (1) that the combustion gases contained no sulphurous, incompletely burned products ; (2) that an improvement in the combustion value of the isoamyl solution was caused by a repeated use of a wick.

An isoamylmercaptan with a theoretical S content of 3·030% gave a value of 2·744% S in a lamp with a new cotton wick, a second estimation without renewing the wick gave 2·862% S, third and fourth estimations gave values of 2·895% S, 2·872% S respectively. The theoretical value of 3·03% was never obtained. On rinsing with 10 ccms. benzine or alcohol the result increased to 2·900%. The correct value was still not obtained when glass wicks were used, as these were found still to contain sulphur.

In conclusion, the desulphurising effect of silica gel on solutions of *higher* aliphatic sulphides in kerosenes is no different from that on diethylsulphide. With diethyl and dipropyl sulphides the effect is the same, but the desulphurising influence of silica gel on dithyldisulphide—and dipropyldisulphide—kerosine solutions is somewhat weaker. The mercaptan solutions give too low a value due to the decomposition of the mercaptans with the formation of sulphur-containing asphaltic substances which are deposited as a brown residue on the wick or glass capillary. W. S. C.

See also Abstracts Nos. 155 and 174.

Fuel Oil.

176. Practical Experience in Firing Refractory Materials with Oil. *Chim. et Ind.*, 47, 2, 282.

From a paper by Mr. Frank West, M.I.Mech.E., who stated that normally the method would prove too expensive, but for a period in 1926 was found useful, and would no doubt prove so again in similar circumstances.

The Derbyshire Silica Firebrick Co., Ltd., at Buxton, manufacture silica refractories from silica bonded with clay, the squares produced being used for blocks for combustion chambers which have to stand temperatures from 1200°—1500° C.

Liquid tar had previously been used, but the heating was so rapid that all the bricks were cracked. However, a mixture of coke breeze and tar, burned like coal, proved quite effective, till the price of tar made it uneconomical.

Oil Firing of Beehive Kilns.—The firemouths of the kilns were built in, leaving only small spaces for the regulation of air, and the burners were placed inside the furnaces, annular space serving for the combustion space, and the setting of the blocks was modified for more efficient circulation of the gases. Larger steam pipes capable of maintaining 30 lb. sq. in. were also used, and the number of burners used was increased towards the end of the second burn.

TABLE A.

Description of kiln.	Total capacity, 3 in. equivalents.	Coal. Weight of coal used per 1000 3 in. equivalents. c. q. lb.	Oil. Weight of oil used per 1000 3 in. equivalents. c. q. lb.	Grog and wasters.
Series of 6 semi-continuous kilns.	36,050	9 3 1 (1093 lb.)	— — —	7·5%
		3 3 23	3 3 8 (428 lb.)	6·0%
		— — —	5 3 0 (644 lb.)	5·6%
Beehives ..	25,760	16 0 22 (1814 lb.)	— — — (1017 lb.)	3·8%
		— — —	9 0 9	2·25%

TABLE B.

Costs of fuel per 1000 squares, 3 in. equivalents.

Description of kiln.	Total weight of fuel. t. c. q. lb.	Rate.	Total cost. £ s. d.	Cost per 1000 3 in. equivalents. £ s. d.
Series of 6 continuous kilns (oil)	17 12 0 0	25s.	22 0 0	0 12 2
	7 2 2 0	25s.	8 18 1	1 1 3
	6 17 2 0	85s.	29 4 5	
	10 8 0 0	85s.	44 4 0	1 4 6
Beehives :—				
Coal.. ..	20 17 0 0	25s.	26 1 0	1 0 3
Oil	11 14 0 0	85s.	49 14 6	1 18 7

Oil Firing in Semi-continuous Kilns.—In a starting kiln of a range of semi-continuous kilns, a burner was placed in each of the furnaces and one at each end of the kiln. A combustion chamber 18 in. wide was constructed by the kiln walls and bags, and a dividing wall built to deflect the flames on to the setting. The two end burners were used for the first 45 hours, when they were removed to the two end fireboxes till the finish of the burn. This arrangement was highly satisfactory and the burners proved easy to control.

In all cases the goods were clean and in good condition.

All over the cost of oil firing is approximately equal to that of coal, but the wear and tear of the kilns using oil appears more favourable than with coal. A. R. S.

177. Treatment of Hydrocarbon Residues and Product Obtained Thereby. U.S.P. 1,660,295. R. T. Goodwin. Feb. 21, 1928.

The pressure fuel obtained from pressure stills is usually unsaleable on account of the carbonaceous material which it contains. By this process, however, a clean oil is obtained.

After the desired amount of pressure distillate is obtained in a cracking process, the pressure tar is run through a cooler to reduce the temperature to about 130° C. It is then mixed with a dilute acid, which may be sulphuric, hydrochloric or acetic acid. The treatment may take place under the working pressure of the plant, and the mixture of oil and acid is run to a setting or thickening device. After a suitable period the tar may be drawn off as a clean product. The remaining portion containing solids may be filtered preferably through a rotary vacuum filter.

Inert granular matter like coke may be added to secure more efficient filtration.

The treated oil is stated to be stable and does not produce substantial amounts of sediment after long standing. R. P.

Lubricating Oil and Wax.

178. Wax and its Manufacture. G. W. Cupit, junr. *The Refiner*, 1928, 7, 1, p 67.

This Paper is a second and concluding article upon the subject. Sweated waxes possessing acid-treating qualities are obtained from first, second and third intermediate scale waxes, and in many cases have to be blended to give the final melting-point desired before acid treating. After blending, the temperature of the sweated wax is raised to 140–150° F. to allow water and dirt to settle out. Time for settling is usually 4–5 hours. Care should be taken not to heat the wax to too high a temperature, as any foreign organic matter present will cause the wax to become unnecessarily dark, requiring excess acid which may or may not remove the colour. After removal of the water and dirt the temperature is raised to 180° F. and the wax is pumped to lead-lined steam-jacketed agitators. When the case is half full, steam is turned on and pumping continued. When full, the temperature must be 180° F. before any acid is added. Agitation is done by air, and as soon as agitation commences 66° Bé sulphuric acid is blown or pumped into the agitator. Usually a dryer of about 3 lb. of acid per barrel is given the wax to ensure the removal of moisture. The time for blowing is 20–30 minutes, after which it is allowed to settle for 45 minutes. The sludge formed is then drawn off and a subsequent pump of 21 lb. per barrel added and the contents of the agitator blown for 45 minutes, then allowed to settle one hour. The sludge is then drawn off.

After removal of all the sludge, 10 per cent. by volume of hot water is pumped into the wash agitator, and this is followed by the wax, neutralising with 5–8° Bé sodium hydroxide but with the omission of air agitation. The wax is then freed of soaps as soon as possible by means of a hot water spray wash, repeating the wash if necessary. After washing, the contents of the agitator are allowed to settle for one or two hours and then the soaps and

water are drawn off. Live jacketed steam is then turned on and the contents air-blown. An average sample of wax is then tested with phenolphthalein, and if slightly alkaline it is ready for a subsequent blow wash with 25% of hot water. If not alkaline, add more caustic. After the wash shut off air and raise temperature to 195° F. Allow to settle for ¾–1 hour and draw down to wax. Allow to settle another hour and draw down to clear wax. Then air blow the wax till bright. The wax should now show a slight pink colour to phenolphthalein.

For filtering, after the bottom canvas has been laid in position and a weight ring placed upon it, the clay chute is opened to allow clay to run into the filter. When charged to 18-inch outage, a top canvas is placed in position and a thin iron weight ring laid on it. The filter is finally charged to one foot outage. The treated sweated wax is then pumped into the filter. After the filter starts running it is shut off at the outlet for 40–45 minutes to allow the clay to become wax soaked. The temperature of the wax entering the filter is 160–170° F., and that leaving the filter 170–180° F. The rate of flow is regulated so that 12 barrels of wax are filtered per hour. The filter is allowed to run till the colour of the wax is so high that it cannot be brought down by blending. This colour range for refined wax is 40–60. The filter is then allowed to drain.

Before the clay can be burned in the rotary kiln it must be washed with naphtha to remove the wax. The filter is given two applications of 50 barrels each of clean 60° Bé naphtha, which is pumped through the filter to a steam still. The clay is then steamed to remove the naphtha, and it is then dried by suction. By steam distillation both naphtha and wax are recovered. The filter is then dumped and the clay regenerated in a rotary kiln. The clay can be burned as many times as it will give a good filtering yield of wax. New clay or No. 1 clay weighs 15,000 lb. per filter. To aid in the filtering properties of burned clay 500 lb. of new clay are added each time the clay is burned, so that No. 10 clay weighs 20,000 lb. Under average conditions new clay will run from 400–600 barrels of wax. The wax is now ready for moulding into slabs for the market. It should meet the following specifications :—

1. It should be odourless and tasteless and remain so.
2. It should be stable to light and heat.
3. It should be within melting-point range.
4. It should not contain oil and should meet the colour test.

S. W. C.

179. Reclaiming Used Lubricating and Insulating Oils. A. F. Meston. Assr. to The De Laval Separator Co., New York. U.S.P. 1,661,731. March 6, 1928.

A process is described for purifying used lubricating and insulating oils, especially turbine, transformer and switch oils which ordinarily contain finely divided solid carbon, water, sludge and organic acids. It consists in heating the oil to a temperature of from 150° F. to 200° F., and then thoroughly mixing with from 2 to 5 per cent. by volume of a solution in water of trisodium phosphate made by dissolving about 15 parts of the salt in 100 parts of water. After agitation, the oil is cooled slowly (2 hours or more) down to a temperature of 120° to 140° F., during which a large part of the water solution, dirt and soaps will settle to the bottom and may be drawn off. The partially cleaned oil is then run through a centrifugal oil purifier and then thoroughly agitated with about one to four per cent. of activated earth, allowed to settle for five to ten hours to remove most of the earth, then finally passed through the same centrifugal. A very efficient earth is made from montmorillonite (a

kind of bentonite from central California) which is finely ground, acid treated and dried. Fuller's earth and other earths will do but are less efficient.

H. S. G.

See also Abstracts Nos. 180, 182, 189, 190 and 198.

Special Products.

180. Reclaiming of "Black Kontact V" on a Manufacturing Scale. B. E. Vlasenko. *Azerheidj. Neft. Khoz.*, 1927, **10**, 86–88.

"Black Kontact V" is the sludge formed during the refining of petroleum jelly. It is placed in lead lined steam kettles which are heated with steam coils agitated by means of air for 15 minutes and allowed to settle at 75° for 8 hours. The acid and water are drawn off, more water added and after agitation of 20 minutes at 85°, the whole allowed to settle for 12 hours. After a third treatment the water only contains traces of acid. By this method 39 per cent. of 67·75 per cent. acid can be reclaimed, while the yield of sulphonic acids amounts to 60 per cent. The reclaimed acid, mixed with 2¼ vols. of fuming acid, gives 92½–93 per cent. acid, which is an excellent refining agent for treating oil, giving a better colour than fresh acid. The sulphonic acids are soluble in water.

S. F. B.

Analysis and Testing.

181. Charcoal Test for Gasoline Content. *Oil and Gas J.*, 1928, 26, 33, 128.

The gas testing committee of the California Natural Gasoline Association have selected the pressure charcoal method on which to standardise, and give the following test as a tentative standard.

A. Gasoline Absorption.

1. *Apparatus.*—A sampling device shall be used which will give a positive sample of the gas to be tested, without air dilution conforming to 3 specified types. For the gas meter, any accurate form of measurement by means of positive, displacement or orifice meter, allowing readings to the nearest hundredth of a cubic foot. The gas drier shall be 250 c.c. capacity and contain granular anhydrous calcium chloride. The charcoal tube shall be ¾ in. I.P. by 30 in. overall length and of 260 c.c. volume, exclusive of fittings. The thermometer shall be graduated in 1° F. and of a range suitable for the temperature encountered. A suitable manometer shall be used for indicating the pressures under which the gas is metered to within an accuracy of 1 in. of water.

2. *Procedure.*—The gas which shall represent a true sample is passed through a scrubber to remove entrained matter. At least 4 cubic feet of gas shall be allowed to pass through the metering device to purge it. Readings shall be taken at cubic foot intervals to determine the average flow termperature and meter pressure. The drier should be placed on the discharge side of the meter. The meter having been by-passed, a sample tube, containing dry charcoal with a minimum of 40 minutes activity by modified chloropicrin test is attached to the outlet of the drier which has been filled with gas to eliminate dead-space error in measurement. A thermometer is placed in the top of the sample tube and immersed in the charcoal to a depth of 2 in. The test is then started

by opening the valve between the sample tube and drier, the flow being adjusted to conform with the desired rate, the maximum rate being as follows :—

Up to 1 gallon/M	15	cubic feet	per hour
From 1 to 3 gallons/M	10	,,	,,
Above 3 gallons/M	6	,,	,,

The termperature rises are recorded and the test stopped at the top of the fourth rise by closing the valve on the outlet of the drier.

B. Distillation.

1. *Apparatus.*—The retort shall be made of copper or steel and be of 1 litre capacity. The condenser bath capacity shall be at least 1,200 cubic inches. The condenser tube to be of nickel-plated steel or brass tubing with an area of at least 100 squares inches, and have a minimum pitch of 1 in. to 8 in length. The receiver capacity shall be 100 c.c. calibrated in half c.c. divisions and shall be completely submerged in a receiver bath of suitable proportions. Thermometers graduated in 2° F. shall be used for indicating the termperature of the condenser and receiver baths, and a low distillation thermometer. A zero 60 lb. pressure gauge graduated in 1 pound intervals shall be connected to the receiver to indicate the distillation pressure. A pressure regulator shall be attached to the vent off the receiver, which shall maintain a back pressure of 30 lbs. The allowable pressure variation shall not be greater than 1 lb.

2. *Procedure.*—125–150 c.c. of glycerin dehydrated to 5 per cent. shall be added to the charcoal in the retort. The temperature of the condenser and receiver baths shall be maintained at 32–44° F. The distillation pressure shall be 30 lbs. No vapour shall be vented through regulator until distillation pressure is reached. The distillation period shall not be less than. 15 minutes from time heat is applied to retort and shall continue until 20 c.c. of glycerin have been distilled and collected in the receiver. The volume of gasoline condensate shall be read at 30 lbs. pressure and 32–34° F., and this apparent volume corrected to 60° F. by means of the expansion factor 0·0008 per degree. For calculating the gallons of gasoline per 1,000 cubic foot, the volume of gas to be used shall be the observed volume corrected to the standard conditions of gas measurement.

C. Charcoal Activation Test.

Air under pressure is bubbled through bottles of concentrated H_2SO_4 and the dried air passed through bottles containing C.P. carbon tetrachloride immersed in an ice bath at 32° F. The saturated air-vapour mixture then passes through the flow meter, then through the charcoal tube and finally into the gas flame in which the copper coil is suspended. The glass charcoal tube should be exactly 14 m.m. in diameter and 10 cm. long. The flow meter is calibrated so that it passes exactly 766 c.c. per minute. The carbon tetrachloride time multiplied by 3 gives the chloropicrin time.

D. Meter Calibration.

Using any of the specified types of provers, tests should be made using rates which will reproduce as nearly as possible the conditions under which the meter is to be used. Adjustments should be made until the meter agrees with the prover within an error of plus or minus 1 per cent. Meters in constant service shall be recalibrated at least once in each two weeks.

S. W. C.

182. Laboratory Apparatus for Testing Filtering Clays. C. F. Barb. *Refiner*, 1928, 7, 1, 82.

The apparatus consists of an electrically heated agitator made of an 18-in. length of 4-in. pipe with a cone bottom discharging through a ¾-in. nipple.

A steel head is welded in the top of the agitator and a ¼-in. pipe runs through the head to the extreme bottom of the cone. This pipe carries the agitating vapour, which may be steam, natural gas, carbon dioxide or air. Steam is satisfactory above 300° F., and air for lower temperatures. Natural gas is the best all-round medium and gives a sweet finished product. The agitator discharges through the ¼-in. nipple at the bottom into a filter chamber made of an 8-in. length of 4-in. pipe with a steel head welded in. An iron gas cock is placed between the filter and the agitator and is closed, while the oil-clay mixture is being agitated. The plate of the filter is an 8-in. cast iron disc with an ¼-in. hole in the centre, and is held against the filter bottom by two ¼-in. bolts. A disc 3¼ in. in diamter is cut out of heavy wire screen and has a tin clip soldered in the centre, which clip fits the ¼-in. hole in the centre of the cast iron disc, and keeps the screen in the centre of the plate. The filter cloth is heavy canvas and fits on top of the screen, but is pinched between the plate and the filter bottom when the units are set up on the holding bolts. The unit has been tested at a working pressure of 80 lbs., though filtering pressures seldom run above 50 lbs. per square inch. The unit is suitable for clay pulp treatment of oils, as well as for dry agitation and filtration.

The method of operation is described. Filtration is carried out by means of air pressure, air being supplied to the agitators after the charge has drained into the filter chamber. The agitator may be charged and operated while the filter is being dumped, so that tests may be run very rapidly.

The apparatus has been used for testing the efficiency of unknown clays by first making up a series of colours from one stock and a good Florida earth, using different quantities of earth per unit volume of oil. These samples are sealed and used as standards for comparison. In running an unknown clay, a ratio of 3 lbs. of clay per gallon of oil is used, and if the resulting oil has a colour closely comparing with that obtained from the standard earth on a 2 lbs. per gallon basis, then the efficiency of the unknown clay is said to be 66 per cent.

S. W. C.

183. A Modified Weighing Pipette. Edgar. *Ind. and Eng. Chem.*, 20–II–158.

The apparatus consists of a Lunge pipette, the bottom receptacle of which, is flattened so as to sit conveniently on the balance pan. The three bulbs are graduated volumetrically and it is claimed that this simplifies the weighing out of constant amounts of liquids as in routine analysis.

A. R. S.

184. The Determination of Oxygen in Organic Compounds. Glockler and Roberts. *J.A.C.S.*, 50–3–828.

The method consists essentially of passing a known quantity of oxygen in a closed circuit over the combustion boat absorbing the carbon dioxide and water formed and calculating the oxygen present in the sample from the observed amount of oxygen used.

Oxygen is made from solid potassium permanganate and the volume measured in a Ramsay burette and from this it passes to the combustion system consisting of the tube containing the boat and platinized asbestos, to which are connected the absorption tube containing P_2O_5 and KOH and a return tube of glass. Between the end of this tube and the other end of the combustion tube is a tubular reservoir of capacity suffiicient to hold the oxygen necessary for the combustion and a Sprengel pump to calculate the oxygen.

A blank is first performed, since it is found that oxygen is absorbed in the absorbtion tube, and then the same oxygen is used in the combustion which

using approximately 30 milligrams of sample, takes 45 minutes, after which sweeping is continued for a further 15 minutes. The apparatus is then brought back to its original state of temperature and pressure, the oxygen measured and the absorbtion tube reweighed.

The recycling of the oxygen seems to ensure complete combustion and copper oxide is not used because of the uncertainty of its returning to its original state on oxidation.

Using benzoic acid the authors obtained results differing in the extreme by −0·87 and +1·93 on the calculated amount of oxygen.

A. R. S.

185. Determination of Sulphur in Volatile Fuels. Kennedy. *Ind. and Eng. Chem.*, 20–II–201.

By the A.S.T.M. lamp method large variations in sulphur content are said to be found by different operators when the fuel contains much carbon disulphide. The following method is designed to overcome this.

The fuel is contained and measured in a reservoir from which it flows through a regulator consisting of an unbored stopcock on the surface of which is etched a tapering channel, which affords easy and accurate regulation. From this the fuel drops on to glass wool, over which heated air regulated by a similar device passes. The vaporisation chamber is heated by a nichrome wire winding of 65 ohms resistance, so that if two lamps are connected in series of a 110-volt system, the necessary temperature is obtained.

The flame used in ½ in. high and should be colourless or slightly yellow and when the fuel reaches the upper mark of the reservoir the lamp is placed beneath the absorber and left without attention; the lower mark is reached when it is withdrawn. Another sample can be analysed as soon as the reservoir empties no washing out being required. The average time for determination is 45 minutes and with 50 samples of 0·04 to 0·17 per cent. sulphur the variation between two runs with one operator was 0·0013 per cent. sulphur.

A. R. S.

186. A Comparison Between the Usual German and Dutch Methods for the Determination of the Volatile Constituents of Coal. D. J. W. Kreulen. *Brennstoff-Chem.*, Vol. 9, No. 1, p. 2.

The modified American method for the estimation of coke from solid fuels was introduced into Holland about two years ago, whilst the usual "Bochumer" method had been employed in Germany, making use of the special crucible introduced by Berthold.

The same process when worked in Germany and in Holland gave values differing by several per cent. This appears to be due to a difference in the mode of heating. In Holland the heating is carried out so that a flame is just extinguished on addition of a supply of air, and is then adjusted to the prescribed height; on the other hand, the heat is applied continuously in Germany. The latter method appears to be more advantageous.

A further reason accounting for the difference in the experimental results is the application of previously-dried coal in the German analysis. Coals containing 1–2 per cent. water are employed in Holland; thus no change in weight due to humidity occurs. In the estimation of volatile constituents the water content is found first and the percentage of volatile products referred to dry coal is calculated by difference.

In order to obtain a "washed product" the water content of the coal is next determined. The coal, which has been already dried in the laboratory is pulverised, further dried for several hours in air, and its water content redetermined. Finally, the values for ash content, volatile products and heat

capacity of the dry substance and of the crude coal are calculated. The "washed product" must be dried in air, not in a drying oven. Kreulen considers it essential to proceed always from air-dried coal in technical analysis.

The following numerical table gives a comparison of the volatile products obtained from eight different types of coal determined according to the individual methods.

TABLE I.

Coal.	Method.			Difference.		
	I.	II.	III.	I.–II.	I.–III.	II.–III.
A	44·71	44·63	43·95	0·1	0·8	0·7
B	40·25	39·65	39·17	0·6	1·1	0·5
C	34·18	33·71	33·50	0·5	0·7	0·2
D	31·56	31·16	31·26	0·4	0·3	=0·1
E	25·13	24·49	24·20	0·6	0·9	0·3
F	21·13	20·66	21·44	0·5	=0·3	=0·6
G	16·60	16·40	15·73	0·2	0·9	0·7
H	9·75	9·22	8·87	0·5	0·9	0·4

Method I. signifies the "modified American method," method II. the "German Bochum process," method III. the Dutch modification of the Bochum process. In all three cases the air-dried substance was employed. a determination of water-vapour content was next determined so as then to be able to calculate the further results from the dry substance. The values obtained in method I. were determined by Kreulen, whereas those obtained in methods II. and III. were determined in the Dutch laboratory in Duisburg.

In conclusion it is evident from the given data that in the determination of the values of volatile products from coal no special difference occurs, regardless of whether one adopts the usual processes worked in Germany or in Holland. It is essential to utilise air-dried coal in both cases always. The values of the volatile products are about 0·4–0·5 per cent. higher in the usual Dutch method than those obtained in the usual German method.

W. S. C.

187. THE ESTIMATION OF ASPHALT IN PARAFFIN OILS. Eng. J. Mzourek, Prague. *Petr. Zeit.*, Vol. 24, No. 9, p. 403.

Mzourek recommends for the estimation of hard asphalts the application of normal benzine at boiling point in preference to the separation with benzine or a mixture of alcohol and ether *in the cold* (Holde's method).

The procedure is as follows: 5 gms. of oil are weighed into an Erlenmeyer flask, dissolved in 40 times the volume of normal benzine and boiled under a reflux condenser for 2–3 hours. Most of the asphalt collects into a ball and settles after about half an hour. When cold the clear oil is filtered and the precipitate washed with benzine in the usual manner and further treated. As a result of several experiments it was found that the filtrate was no longer turbid after several days.

This analysis is quicker than the usual separation in the cold. The accuracy of the process has been tested with different oils and a comparison of results of the usual cold precipitation and subsequent extraction of waxy hydrocarbons with light benzine (50–65° C.) (S. Prozynski's process), and of the hot precipitation process is given. It appears that in this modification of the asphalt estimation, the result is not influenced so strongly by the constitution of the benzine as in the cold precipitation.

W. S. C.

188. THE VARIATION OF THE VISCOSITIES OF TARS WITH TEMPERATURE. H. M. Spiers. *Brennstoff-Chem.*, Vol. 9, No. 5, p. 77.

The work of H. Mallison and F. Soltau in a previous publication on the application of the formula log. $C_1/C_2 = K(T_2 - T_1)$ to show the change of viscosity of tars with temperature is briefly described.

Experiments were carried out with a temperature range in the first instance of 9–19° C. and 18–29° C. and secondly with a temperature range of 19–27° C. and 29–37° C. As a result of these experiments it was found that a definite physical change of the tar occurred at a definite point with decrease in temperature, and at this point a sudden deviation from the straight line was shown in the graph; however, a direct relationship still existed although the values of the constants varied. The physical change in the nature of the tar may be attributed to different causes, e.g. crystallisation of naphthalene, separation of paraffin, or a change in the dispersion state of the colloid.

Spiers has observed cases in which the relation between the temperature and the logarithm of the viscosity is represented by two and often three straight lines, which appear above the temperature range at which the experiments with the individual tars have been performed. When the "turning point" of the curve between the two temperatures at which the measurements were carried out, has been found, the value of the viscosity referred to normal temperature can be determined with sufficent accuracy.

The relation log. $C_1/C_2 = K(T_2 - T_1)$, showing the dependence of temperature on the viscosity of a tar, is accurate within a temperature range of 10° C. (=18° F.). The value of K is found to change suddenly at a definite temperature, which depends on the nature of the tar, and this point indicates a sudden alteration in the physical properties of the tar.

W. S. C.

See also Abstracts Nos. 157, 175 and 209.

Refinery Plant.

189. STEAM TEMPERATURE REGULATION IN DISTILLATION PROCESSES AND IN THE MANUFACTURE OF PARAFFIN. Director Eng. E. Belami, Villach. *Petr. Zeit.*, Vol. 24, No. 5, p. 199.

This article deals with the importance of steam temperature control in distillation processes in mineral oil work utilising the Foster Tube Still and the Benson boiler.

The Benson high pressure boiler is fired with oil and its steam is sent at 200 atmospheres pressure and at 420° C. directly into a high pressure turbine. The oil is passed through steam pumps directly from the crude oil boilers into the Foster Tube Still and heated to 758° F. = 400° C. The outlet of the Foster Tubes terminates in an expansion drum into which the residue is drained while the vapours are passed into fractionating columns. The oil in the crude oil boiler is pre-heated by steam to 475° F. (246° C.), this accelerating the distillation process.

The Foster Tube Still consists of 308 tubes carrying oil, which are connected to 308 tubes, which convey the vapour. The material pumped from the tubes is a 27° Bé product. The residue has a gravity of 16° Bé and a cold test of 25° F. = 4° C.

The following points are essential for an efficient steam regulator:—

1. Uniformity of outflow temperature with fluctuating inflow temperatures.
2. Variations in the steam which occur suddenly must not affect the stability of the outflow temperature.

3. The outflow temperature must be capable of change at any time.
4. The cooler is not allowed to consume more than the theoretical quantity of water.
5. The quantity of cooling water must be capable of adjustment by hand, if necessary.
6. If, in this case, too much cooling water passes out, this must be removed as quickly as possible from the regulator.
7. The regulator must be immediately adjusted to all fluctuations in temperature, pressure, and quantities of steam.
8. No decrease in pressure must occur in the regulator.
9. The inflow and outflow temperature of the steam must always be controllable.

Two diagrams of the boiler and still are given.

The regulator is so constructed that a complete evaporation of the injected water is guaranteed and no loss of water occurs. A loss of heat rarely takes place, since the heat required to evaporate the cooling water is recovered entirely in the form of steam.

This apparatus should be utilised in mineral oil refineries and in the manufacture of paraffin where steam is usefully applied.

W. S. C.

190. Distillation with Modern Pipe Still. H. S. Bell. *Oil and Gas J.*, 1928, 26, 41, 99.

Theoretically, if oil is heated by a single flash in a pipe still, the partial pressure effect of the light products would result in the removal of the same amount of vapours at a lower temperature than that required in a shell still. Practically, we find that because of the necessarily extensive fractionating equipment required when separating many products all in vapour phase, the oil has to enter the separator at temperatures corresponding to, or even above, those required in the batch still. But in the case of shell stills, oil will not absorb more than 3 to 4 B.T.U. per square foot per hour per degree difference between flue gas and liquid unless some means of forced circulation is employed. Analysis of heat input into oil passing through tubes at high velocity show that 12 to 14 B.T.U. per hour can be safely transferred for each square foot per degree temperature difference. Counter-flow, velocity and intimate contact are essential to input of heat, and here the tube still lends itself to efficient design. In order to obtain maximum transfer, turbulent flow should be maintained throughout the coil.

From the outlet of the pipe heater the mixed oil vapours are introduced to an evaporating chamber. There are many different types in service. Some are horizontal with longitudinal splash plates, some vertical with conical baffles. Others introduce the oil at a tangent in a vertical separator, whilst sometimes the stream is led directly into the fractionating column. The evaporator should spread the oil out in a film to allow plenty of disengaging surface and be of a size to give sufficient time for separation. Here is accumulated a large volume of vapour composed of all the overhead products and these have to be separated into close commercial cuts. The bubble cap tower has superseded all other types for this service in connection with modern pipe stills. The liquid descending through the tower is supplied by returning a portion of the condensed vapours, leaving at the top. If fuel is expensive, the column should have more plates and less reflux than would be the case with cheap fuel.

Correct furnace design is of prime importance to successful pipe-still operaton. A surface will absorb heat from a radiating surface at about 10 times the rate of the absorption of heat by convection. Therefore the part of

the pipe still doing the most work per square foot is that exposed to direct radiation. The design of this section must carefully analyse the oil temperature and velocity to be sure that it will be able to absorb this heat, otherwise carbonisation and burned tubes are inevitable. Radiant heat and flame impingement on the same surface create a serious condition. By elimination of the flame impingement higher furnace temperatures may be carried. The radiant tunnel construction is a development along this line. Another line of approach is to keep the furnace temperatures at a lower degree. The admission of excess air to accomplish this is wasteful from all standpoints. Recirculation of the flue gases will attain the results desired, and at the same time maintain a high efficiency. Further, the suspension of tubes by a structure independent of the furnace brickwork eliminates cracking due to expansion strain with consequent air infiltration and decrease of efficiency. The usual sizes for the tubes are 3 and 4 inches, and if the use of such sizes results in too much pressure drop, it is considered good practice to use two or more tubes in parallel.

The control of the bubble towers is important. The inlet temperature at the base is capable of control by the heater and the top temperature will depend upon the amout of reflux. The reflux is furnished either by using partial condensers or by pumping back a portion of the finished top product, Both methods give the same results. It is customary to use oil for cooling, thus making the partial condenser part of the exchanging system. As corrosion is worst at the condensing points of products ranging from 180°–250° F., relatively high maintenance may be expected for the partial condensers.

Electric and air-actuated controllers are in use with both systems to control the top temperature. The ideal tower control is one that gives a balanced condition of the reflux so that the tower temperature is held constant. Undoubtedly a pyrometer is more sensitive than any other type of temperature measuring instrument. However, there must be a periodic make and break in a pyrometer temperature controller, and during the period that the thermocouple is disconnected from the motor-operated valve the reflux may become unbalanced, with the result that the tower temperature will not remain constant. Its advantages are that it may be located at any desired point without affecting its sensitivity and the control may be set at the exact temperature at which the operator desires to control the tower. Air-actuated controls may be mercury-filled, vapour-tension and gas-filled instruments. As a class, the advantage is that as soon as the temperature is transmitted to the regulator the reflux is brought into balance with the tower conditions. The mercury-filled instrument is the most sensitive, but requires short lengths of tubing, and therefore location near the point of control. The vapour-tension instrument is limited to a smaller range of settings, but its location may be more variable. The gas-filled instrument is not as sensitive as either of the others, but lends itself to greater flexibility in that it may have as long a capillary tubing as the vapour tension, and may be of the direct-set type operating equally well over a wide range of temperature settings.

The difficulty of obtaining side-streams to specifications is noticeable; the overlap of distillation curves of adjacent plates is excessive and effective separation is possible only between the top and bottom products of any one tower. This is, in effect, an argument in favour of multiple towers. The greater the difference in boiling points between two products the easier is the separation in any given tower. Thus the separation of gasoline from kerosene is easier than that of kerosene from gas oil. The further we go into the crude the more difficult is the separation of successful cuts. When we come to the fractionation of the overhead products from a crude being run

to cylinder stock, the separation of the heavy ends is very important. If any wax distillate or gas oil is left in the cylinder stock, the flash, fire and viscosity relationships are altered and the dewaxing problem is complicated. Likewise, the presence of cylinder stock in wax distillate is detrimental to wax removal by cold pressing. In the present state of the art, it is best to remove only a portion of the distillate in the once-through pipe still and then use other apparatus. Batch stills or circulating pipe stills can be used, and the series pipe still also appears to have possibilities.

In general pipe stills may be divided into three distinct types: (1) the "once-through" or single flash; (2) the series pipe still, where two or more are operated in series; (3) the circulating pipe still.

The first is the most popular and has the broadest field. With a properly designed single column or multiple towers, separations to meet specifications of all light products can be made with excellent yields and cost figures. For heavier products the separations are satisfactory as far as gas oil and fuel oil are concerned. It appears to have limitations when cylinders stock is the desired residual product.

The second type presents two advantages. Here the volume of cuts removed in vapour phase is greater than in the case of the single-flash unit, therefore, the possibilities of vapour heat exchange are greater. Secondly, if close separation of residuum from the next heaviest product is desired, a second simple tower will give better results.

The circulating type of pipe still is in reality a batch still with an independent means of supplying the heat. As the oil increases in temperature, flue-gas temperatures must also rise, accounting for the poor efficiency, as compared to the once-through or single-flash unit. One advantage is inherent in any batch operation, and that is close control of the residual product. A diagram for an improved design is given, the advantages being close control of bottoms coupled with good fractionation and over-all fuel efficiency. Its disadvantages are a higher steam consumption and limitations as to size, the maximum for one unit being about 1,500 bbls. daily capacity.

A description of several commercial pipe stills in actual operation is given, the plants being chosen as typical examples.

S. W. C.

191. IDEAL DISTILLATION UNIT IN REFINING. A. G. Peterkin. *Oil and Gas J.*, 1928, 26, 3, P. 64.

In the past, heat applied either directly or in the form of steam has been the major part of distillation costs, by reason of furnace limitations and of the incompleteness of separation in any one distillation operation. How close we may come to the ideal depends so far as heat input is concerned on the type of furnace; in regard to heat recovery and separation, on the fractionating equipment. The article deals with the single-flash method of distillation by a pipe still. The furnace was designed to burn oil. The tubes, 4 in. outside diameter, from two continuous passes, joined at the inlet and outlet of the still. A steam superheater is inserted in the convection section. The heat input was found to be 78 per cent., showing that the pipe still has taken us far along the road to the ideal, at any rate so far as heat input is concerned. As regards tube arrangements, the merits of the single passage are obvious and the objections equally so. The furnace pressure drop varies with the throughput and to a surprising extent with the volatility of the charge. The one-pass furnace limits the maximum practical capacity; the twin-pass doubles capacity for equal pressure drops, but because of uncertainty in division of the stream, increases the danger of coking, since both passes are subjected to not widely differing heat conditions. Experience

shows that the difficulty of maintaining the same outlet temperature on both passes increases as the pressure drop decreases, and also as the average volatility decreases. The question of safe velocity in the tubes from a coking standpoint has sometimes been discussed in terms of turbulent liquid flow. Such flow in this type of distillation occurs only in regions of safe temperature. Pressed wax distillate run at 730° gave a comparatively low inlet pressure and showed an alarming tendency to distribute unevenly. Throttling the inlets on both passes corrected the conditions and avoided coking. It appears that serious coking will not occur when the vapour velocity is sufficient to keep the liquid suspended in the vapour stream and travelling at the same speed, and conversely the liquid may coke if it falls out and flows comparatively slowly along the tube surface. The two-pass furnace may then be limited in its ability to handle crudes requiring high temperatures for the desired overheat separation and containing relatively large amounts of liquid not volatile at the temperature. The most important factor in maintaining constant oil outlet temperatures is the ability to hold constant the quantity and temperature of the heat delivered. The heat recovery from the fractionating equipment and the furnace efficiency are to some extent interdependent. Of the additional heat recovered by raising the inlet temperature of the oil from 400° to 500° F. some 50 per cent. is lost by the consequent increase in stack gas temperature.

In the bubble tower the crude flows through exchangers, being heated, first, by the naphtha vapours, and then by each side stream successively. By this means a heat recovery of about 38 per cent. is effected. If the crude is routed differently, removing now the heat of vaporisation not only from naphtha vapours, but from the vapours as they enter the plates from which the side streams are taken, it is possible to recover 49 per cent. of the heat. Compensating for increased stack losses this might make the comparative figures 38 : 44. Against this is the fractional consideration that each side stream cooler must be made sufficiently oversize to allow of the estimated variations in yields. A graph shows in approximation the change in volume of hydrocarbon vapours and of steam based on 1,000 lb. of steam per gallon of crude having been passed through the bottoms all the heat of vaporisation and the heat of the steam removed at the top in one case and through the column in the other. The steam quite clearly takes up much valuable space in the tower above the vapour intake, even if used in much smaller quantities and renders no service in return, its work having been done in the stripping section below. At the cost of some complication it may be removed by scaling off the section of the tower below the vapour inlet and condensing it separately, pumping the oil condensed back into the tower at a suitable place. The overhead products are not changed by this method.

Observation of a bubble tray at work is convincing evidence that the interchange is not solely a question of a distorted vapour bubble offering a maximum surface in passing through a pool of liquid; rather of a dense shower of drops suspended in the vapour stream. If we had a tower in which the liquid on each plate is almost entirely in the form of suspended drops, the plate above protected from entrainment, then the diameter might be governed by the liquid flow capacity. The flash of side streams may be markedly improved with minute amounts of steam if applied either in or directly leaving the column. The boiling range of the strippings is surprisingly wide, covering almost the entire range of the crude.

Summing up, then, the single-flash method coupled with single tower and overhead removal of vapour heat can be used to evaporate crude oil to two 10 per cent. bottoms at furnace efficiencies of 75 per cent. and heat

recovery of 30 per cent. The quality of the naphtha is governed by the top of the tower temperature. The entire system calls for only two points of control, the temperature of the oil leaving the furnace and that of the vapours leaving the bubble tower. The still is extremely flexible and will process a wide variety of raw materials. The yields of wax distillate are large compared with those from coking stills. On the one hand, practically no cracking has taken place; on the other, the separation from high boiling amorphous wax is more complete. The ease of pressing a distillate depends not upon whether it has been cracked, but upon its boiling range, particularly upon the temperature at which the last 5 per cent. boils. Experiments show that, first, a gradual and then a sudden decrease in pressing rate may be expected as the temperature at the 95 per cent. point of a vacuum assay increases, and this in spite of the fact that sufficient gas oil may be added to the lower end of the boiling range to make the viscosities in each case the same.

The only competition of the pipe still, and by no means an assured one, is the high vacuum shell still for the production of lubricating oils.

S. W. C.

192. Distillation Methods and Processes. F. C. Koch. *Oil and Gas J.*, 1928, 26, 41, 109.

The advantages offered in using two pipe stills where 90 per cent. of the crude is to be removed are:—

1. The straight-run gasoline and kerosene are removed at the lowest possible temperature thereby requiring a minimum of treatment.
2. The cracked naphtha, of which there is a high yield due to the high temperature of the gas oil distillation, is kept separate from the straight run product.
3. The towers required are small although more in number than in other methods.
4. The second pipe still should be able to be shut down without materially interfering with the operation of the other.

Where the distillation is carried out with one pipe still and one less tower the temperatures are lower than in the previous case, which makes it preferable when producing lubricating stocks. This plant is easier to run from all points of view, and it will not be necessary to clean it as often as the second one in case one. A single tower installation requires the tower to be of the same diameter all the way up, since the vapour load at both top and bottom does not vary widely. It is impossible to take off a considerable percentage overhead in this manner, since the temperatures at the bottom will become so high that coking will result. Pipe stills are also capable of producing very close-cut products, such as special naphthas direct from the crude.

The use of towers in conjunction with shell stills is next considered. Various arrangements are given with their uses and limitations briefly mentioned.

S. W. C.

193. Process for Breaking Petroleum Emulsions. M. de Groote and W. C. Adams. (*a*) U.S.P. 1,659,993, (*b*) U.S.P. 1,659,994, (*c*) U.S.P. 1,659,995, (*d*) U.S.P. 1,659,996, (*e*) U.S.P. 1,659,997. E. H. Keiser, (*f*) U.S.P. 1,659,998. C. H. M. Roberts, (*g*) U.S.P. 1,659,999. M. de Groot and B. Keiser, (*h*) U.S.P. 1,660,000, (*i*) U.S.P. 1,660,001, (*k*) U.S.P. 1,660,002. M. de Groote, (*l*) U.S.P. 1,660,003, (*m*) U.S.P. 1,660,004, (*n*) U.S.P. 1,660,005. Date of issue of the above, Feb. 21, 1928. All assigned to Wm. S. Barnichel and Co., Webster Groves, Missouri.

The above inventions relate to the treatment of emulsions of mineral oil and water, such as petroleum emulsions for the purpose of separation. Reference

is made to U.S.P. Serial No. 59,954, Oct. 1, 1925, in which emulsions are subjected to the action of a treating agent consisting of a mixture comprising a condensation product containing a sulpho-aromatic soap forming acid in which the sulphonic group is joined directly to the non-carboxyl end of the aliphatic hydrocarbon chain.

In the manufacture of the above treating agent on a commercial scale a mechanical mixture thereof with certain complex sulpho-aromatic soap-forming bodies is formed, which, while lacking the distinguishing characteristics of the treating agent contemplated, are of equal or greater value as agents for treating petroleum emulsions. In view of the difficulty in analysing such complex organic bodies to determine their chemical constitution, the treating agent contemplated is defined in the above series of patents according to the process employed to produce such agent.

By the expression "soap-forming body" is meant those organic groups which combine with soluble hydrates to give soap-like detergents, and includes fatty substances such as ricinoleic acid, hydroxylated resinous substances, and hydroxylated naphthenic acid such as found in petroleum sludges. The aromatic substances employed may be one or more of the common coal-tar substances, such as benzene, toluene, zylene, cresol, phenol, naphthalene, naphthol, anthracene, tetralin, etc. Mono- or poly-sulphonic acids are equally suitable for the process.

The treating agent is brought into contact with an emulsion in any convenient manner and then allowed to stand in a quiescent state at a suitable temperature to permit separation; or the mixture may be passed through any of the usual separating devices, or heated with or without the addition of water.

The preferred examples of treating agents are as follows:—

(*a*) Phenol-mono-sulphonate and hydroxystearic acid in molecular proportions are subjected to the condensing action of a suitable drying agent such as sulphonic acid.

(*b*) Two molecular parts of phenol and one molecular part of oleic acid are subjected to the action of any sulphonating agent such as sulphuric acid with subsequent separation of excess acid.

(*c*) Oleine is mixed with a molecular proportion of an aromatic hydrocarbon such as phenol or cresol and sulphonated preferably at a temperature below 20° C.

(*d*) Ricinoleic acid is heated with one or more parts of napthalene sulphonic acid until condensation has taken place.

(*e*) Naphthalene sulphonic acid and castor oil are heated with a mixture of oleic acid and hydroxystearic acid until combined.

The above five products may be used in the form of their water soluble alkaline salts, or as their water insoluble salts of the alkaline earths or as esters; *e.g.*, methyl or ethyl esters.

(*f*) Commercial red oil is sulphonated and mixed with sulphonated olive oil in such a ratio that the resulting compound is composed of the following ingredients mixed in the following approximate proportions: viz:—

Stearolactone, 15—25 per cent.
Sulpholeic acid, 15—23 per cent.
Oleic acid, 32—42 per cent.
Hydroxystearic acid, 16—9 per cent.
Stearic acid, 6—1 per cent.
Non-saponifiable, 5—0 per cent.
Hydrocarbons, glycerine, etc., 6—0 per cent.
Water, 5—0 per cent.

The mixture is then partially saponified by adding approximately 8 per cent. of ammonia.

(*g*) A Twitchell type of reagent is first prepared by sulphonating an unsaponified body such as oleic acid, olive oil or castor oil mixed with an equal proportion of tar oil to produce a compound of approximately the following composition, viz. :—

Unsulphonated fatty material, 40 parts.
Phenol sulphonic acid, 5—10 parts.
Non-sulphonated aromatic hydrocarbons, 30—40 parts.
Phenol, 12—18 parts.

Phenol fatty sulphonates less than 10 parts. Approximately 75 gals. of the above compound is then mixed with 37½ gals. of preferably red oil, and the mixture combined with approximately 18¾ gals. of tar acid oil, tetralin, etc., 28 gals. of denatured alcohol or methyl alcohol and 20 gals. of aqeuous ammonia or other suitable base.

(*h*) Castor oil bodies modified by methods as outlined above mixed with 10—35 per cent. cresylic acid.

(*i*) The fatty acid, salt or ester of a modified oleic acid containing not less than 8 per cent. sulpho-oleic acid mixed with 10—35 per cent. cresol or cresylic acid.

(*k*) The fatty acid. salt or ester of modified bodies obtained by sulphonating red oil, olive oil or similar bodies to produce modified oleic bodies containing not less than 15 per cent. of hydroxystearic acid mixed with 10—35 per cent. cresol or cresylic acid.

(*l*) The product obtained by condensing by means of sulphuric acid an organic fatty body such as oleic or ricinoleic acids and a non-hydroxy-monocyclic aromatic body such as benzene, and used in the form of acid, salt or ester.

(*m*) The fatty acid salt or ester of a sulphonated petroleum body derived from crude petroleum by refining or in any other manner mixed with 10—35 per cent. cresol or cresylic acid.

(*n*) The product obtained by heating at 120° C. molecular quantities of petroleum acids derived from the refining of petroleum products and castor oil, in the presence of 3—10 per cent. fatty or aromatic sulphonic acids.

H. S. G.

194. Process of Separating Liquids.—O. L. Barnebey. Assr. to American Solvent Recovery Corporation. U.S.P. 1,661,403. Mar. 6, 1928.

The invention relates to a method of separating the constituents of liquid mixtures in which distillation is supplemented by selective absorption. Suitable apparatus consists of a steam heated still connected by a pipe to the bottom of the first of a plurality of absorbing columns connected in series. These columns are similar in construction and are divided internally by transverse foraminous diaphragms into three chambers. In the middle chamber is arranged a heating and cooling coil embedded in a mass of absorbent material such as activated charcoal. The top of each column is connected by a pipe with the bottom of the next column of the series and the last column is connected to a condenser. Each column is also provided with a vapour discharge pipe leading to suitable condensers. In carrying out the process, the liquid mixture of chemical compounds is heated in the still to cause the vapours to pass over and through the series of absorbing columns, which are preferably maintained at different temperatures regulated by the amount of heating or cooling medium passing through the coils. Generally the series of absorbing towers are maintained at successively lower temperatures, the last column being held at a temperature just

sufficiently elevated so that the vapour of least absorbing affinity is not absorbed but passes over to the condenser. Other vapours are absorbed in the columns in order of their absorbing affinity. When absorption has been carried on to saturation or otherwise, communication between the columns is shut off and the absorbent material of each column is heated causing an evolution of absorbed compound in the form of vapour which is recovered from the respective condenser. H. S. G.

195. Process and Apparatus for Fractionating Hydrocarbon Oils. —J. C. Black. U.S.P. 1,652,886. Dec. 13, 1927.

The invention provides for a process for the production of a mixture of hydrocarbons of predetermined boiling-points which comprises volatilising the hydrocarbons, passing them through a wash tower, washing the vapours with hydrocarbon oil to partially condense them, cooling the condensates, including the wash oil, passing the residual vapours from the wash tower to a second tower and there partially condensing them, utilising the cooled wash oil to further partially condense the vapours in the second tower. At the same time the condensed hydrocarbons are reheated in the second tower to partially volatilise them, the vapours mixing with the other uncondensed vapours therein and finally cooling the ultimate residual vapours to produce the mixture of hydrocarbon oil of the predetermined boiling points. S. W. C.

196. Evaporator and Method of Operation.—C. F. Braun. U.S.P. 1,662,142. Mar. 13, 1928.

The object of the invention is to provide an evaporator or still within which hydrocarbon liquids will be readily evaporated by a method providing a maximum progressive distillation of the various fractions of oil and at the same time a minimum condensation after distillation. The apparatus consists of a tubular vertical still suspended from the top of which is a tube nest. Steam flows upward in the still and around the tubes while simultaneously hot oil flows internally down the tubes maintaining a high surface temperature on the tubes down the outside of which oil to be distilled flows and is evaporated, the vapours passing on with the steam. H. S. G.

197. Process and Apparatus for Distilling Oil.—H. L. Doherty, Assr. to Heat Treating Co., New York. U.S.P. 1,662,105. Mar. 13, 1928.

A process and apparatus are described, by which a mixture of liquids of different boiling points are separated in one operation into a series of fractions having definite narrow boiling point limits and substantially free from substances having boiling points outside these limits. Crude petroleum or other liquid to be treated is supplied to a distilling apparatus through a supply pipe entering a distilling chamber near the upper end of the apparatus. Passing into the chamber, the oil spreads out over the bottom and is heated by contact with a heat-transferring gas passing upwardly through the oil. From this chamber the oil flows through an orifice pipe to a lower vaporising chamber in which it is heated to a higher temperature by contact with hot gas. In this manner the oil flows in succession through a number of vaporising chambers in the lowermost of which the oil is in contact with gas at the highest temperature and the highest boiling constituents are vaporised and absorbed in the circulating gas. From the final chamber the oil flows through an outlet pipe to a series of heat interchangers in which it is cooled to progressively lower temperatures by an upwardly passing current of gas. From the lowest vaporising chamber the gas and vapours enter a condensing chamber and are uniformly distributed

by means of a gas-distributing plate, and pass directly into contact with a cooling coil extending throughout the chamber a short distance above the distributing plate. The temperature of the cooling coil is maintained slightly below that of the vaporising chamber so that the greater part of the vapours will be condensed. To facilitate heat transfer between the cooling medium and the gases a body of liquid condensate is maintained in contact with the cooling coil through which the gas is bubbled. From the condenser the partly-cooled gases pass into the vaporising chamber next above and serve to heat the body of liquid in that chamber. The gas vaporises and absorbs constituents of the oil which volatilise at a lower temperature than that in the vaporisation chamber below, and at the same time any heavy vapours which may have passed through the condenser are condensed by contact with cooler liquid and subsequently returned to the vaporising chamber below. The gas with absorbed vapours passes into a condenser positioned immediately above, and the above cycle repeats itself for each vaporising chamber until in the final condenser the gases are cooled to the lowest temperature obtainable and all the volatile constituents condensed out.

The cooled gas is withdrawn and forced by a fan to the heat interchangers at the bottom of the still and is heated by successive contacts with progressively warmer liquid. From the uppermost heat interchanger the gas passes into a chamber and is raised to a temperature above that of the lowest vaporising chamber by the admission of the hot products of combustion from a furnace forming part of this chamber, and then circulated through the vaporising chambers. The cooled oil residue is withdrawn from the lowermost heat interchanger and forced by means of a pump to the cooling coil in the uppermost condenser, and passes successively through the cooling coils in each condensing chamber, and passes from the final cooling chamber into the final vaporising chamber again and thence into the heat interchangers from which excess liquid is removed from the circuit.

The exhaust gases and oil residues leave the apparatus at nearly the same temperature at which they enter. Radiation losses are reduced by suitable insulation, and accordingly a very small amount of heat need be supplied by the furnace, and the thermal efficiency of the process is very high.

H. S. G.

198. Reclaiming Used Crank-case Oil.—W. H. Ellerington. U.S.P. 1,662,701. Mar. 13, 1928.

The patent describes a process for removing carbon, dust, grit, sand, metal particles and other impurities from used lubricating oil. It comprises intimately mixing lubricating oil containing such impurities with a mixture of soap, sodium silicate and talc, allowing to separate and removing the cleansed oil which may be heated to remove volatile impurities. The soap is preferably composed of palm or cottonseed oil 3 lb., caustic soda 94 per cent. 8 oz., water 16 oz. To this is added sodium silicate 8 oz. and talc powder 8 oz. To the above sufficient water is added as convenient, and the above quantity is sufficient to treat 45 gallons of used oil. It is advisable to first add to the oil about two or three ounces of aqua ammonia, then the above mixture.

H. S. G.

199. Distillation of Petroleum Oils.—R. W. Hanna and R. J. Hanna, Assrs. to Standard Oil Co. U.S.P. 1,653,431. Dec. 20, 1927.

The invention provides for a process for continuously distilling a petroleum oil comprising continuously vaporising the oil, continuously passing the evolved vapours through a three-stage vapour-separating system, in the first stage of which the vapours are subjected to the condensing action of

the feed oil to the system and the condensate from the other stages; in the second stage the vapours are subjected only to the condensing action of the condensate from the third stage, and in the third stage the vapours are brought to a desired discharge temperature by controllably dissipating heat thereof to a separate liquid cooling medium the temperature of which is controlled without varying the character of the feed oil maintained at its boiling point; the feed oil, together with the condensate from such vapour-separating system being passed to the source of evolved vapours, and condensing as one product the vapours from such vapour-separating system. This process is characterised by the separation of no intermediate product. S. W. C.

200. Apparatus for Treating Oil. F. A. Kormann, Assr. to United Refineries Co., New York. U.S.P. 1,661,804. Mar. 6, 1928.

The invention consists of an apparatus for obtaining increased yields of gasoline from crude or heavy gravity mineral oils, embodying a still of conventional cylindrical form with dome and vapour line to a condenser. A metal supporting frame of angle iron is placed in the lower part of the still, leaving a space below for the location of pipes and for the accommodation of a body of oil to facilitate changing and drainage, as well as to prevent injury to the still shell. On this supporting frame high absorbent bricks are placed so as to break joints in successive layers and to fill the still to a point slightly below the liquid level when the still is fully charged with oil. The still is mounted on a furnace setting in which is embodied steam superheaters. Steam is supplied to the superheater from any convenient source and passes from this to a coil in the bottom of the still from which it can be exhausted to the atmosphere or sent along branch pipes and discharged in a finely divided form in the bottom of the still below the absorbent brick or else in the body of the absorbent brick. The still is provided with residua drain pipe, air valve and manholes.

Preliminary to the admission of oil, the still is brought to a temperature of 550°—600° F. by passing steam through the heating coil to the atmosphere. Oil is then charged and is absorbed by the hot porous bricks accompanied by a yield of lighter distillates. After distillation has commenced steam is admitted to the still through the finely perforated pipes either below the absorbent bricks or in the bricks or both. The absorbent material holds the products in finely divided or filamentary form in such intimate relation that reaction takes place, resembling vapour phase crocking, resulting in largely increased yields of the more volatile products. When the condensate coming over has reached a point where the boiling point is above that of the desired product, the residue is drawn off through the drain pipe and the above operations repeated on a new charge of oil.

H. S. G.

201. Distillation of Hydrocarbons. M. R. Meacham, Assr. to Standard Oil Development Co. U.S.P. 1,661,189. Mar. 6, 1928.

An improved method is described of distilling hydrocarbons with steam in an apparatus in which hot oil is carried from the bottom of the still and distributed through a rising current of steam so as to obtain widespread and repeated contact. The apparatus comprises a shell-type still in which, projecting into the top and on down to the header connections, are steam supply pipes. From the header distributing pipes, with perforations directed towards the bottom of the still, extend longitudinally. A dome at one end of the still has the usual vapour-offtake pipe, and arranged beneath the dome is a circulating device consisting of an open-ended tube, the lower

end being near the bottom of the still and the upper end extending just into the dome. Under the tube and discharging into it is a steam jet pipe. Above the tube is a hood deflector and in relation to this, so as to divert liquid downwardly, are deflector plates. A trough extends horizontally along the still under the deflector plates to catch the liquid and distribute it through overflow feed edges throughout the still. Beneath the trough are spaced baffles to receive in progressive succession the liquid overflowing from the sides of the trough and cascade it through the upper portion of the vapour space of the still. With the still operating, the oil being heated and thoroughly agitated by the steam admitted into the inlet pipes, vapours are carried up and the bubbles of steam traversing the oil escape at its upper surface and proceed toward the dome. At the same time steam supplied through the jet pipe raises quantities of hot oil from the lower portion of the still, splashing it out over the deflector plates, from whence it runs into the trough and is distributed in thin cascades on to the baffle plates by which means it is brought into intimate contact with the ascending steam.

H. S. G.

202. Process for Treating Emulsified Oils. C. C. Monger. U.S.P. 1,660,230. Feb. 21, 1928.

Emulsified oil is heated to a high temperature under pressure sufficient to prevent any vaporisation of the oil while in the heater, and by manipulation of a valve the oil is allowed to discharge with great velocity against a solid baffle and into the hot water contained in a washing tank. This sudden release of pressure on the heated oil causes rapid expansion of the globules and as the globules impinge with great velocity against the solid baffle in the washing tank, each globule is forcibly disrupted both by its internal pressure and the physical contact against the baffle. As the globules are broken the water sediment is taken up by the water contained in the tank and the oil is sprayed in a very thin film downwardly assuring a complete washing therein. The oil collecting at the top of the washing tank is free from all water, sediment and foreign matter, and is in a refined state ready for usage.

H. S. G.

203. Process for Separating Oils. J. K. Pierce. U.S.P. 1,660,235. Feb. 21, 1928.

The object of the invention is to provide an expeditious process and compact apparatus to separate water and bottom settlings from oil by chemical means.

The apparatus comprises two tanks appropriately closed to be able to withstand pressure. Untreated oil is supplied to the initial tank through a pipe with minute perforations directed upwardly towards a perforated baffle plate extending horizontally across the tank. The tank contains a chemical solution which is circulated by convection currents generated by a heater in circuit with a pipe leading from near the bottom of the tank to about the middle. The heating circuit is opposed to the tendency of the introduced oil to rise and thus causes the maximum degree of separation.

In the initial tank the lighter oil fraction ascends to the top of the tank and is drawn off through a valve. The bottom settlings form an intermediate stratum heavier than the oil but lighter than the water and chemical solution, and are drawn off from the preliminary tank and pass into the final tank through a trap which eliminates any water and chemical solution. The bottom settlings flow into the final tank where a stronger chemical solution is maintained and continually circulated as in the initial tank through a heater circuit which in this case contains a reservoir for adding chemical solution so that more can be added from time to time to keep up the strength.

An overflow is connected to the tank at the level desired for the chemical solution, and a pump is connected to the overflow pipe in order to withdraw the weakened solution after use in the final tank and return it to the initial tank. The purified oil is drawn off from the top of the tank through a control valve.

Any chemical that will break down an emulsion and permit separation of the oil and water globules is suitable for use in the above process.

H. S. G.

See also Abstracts Nos. 168, 169, 177, 178, 206 and 208.

Engineering.

204. The "Paragon" Thermo-Electric Locomotive. Capt. William P. Durtnall. *Petr. Times*, 1928, **19**, 533–536, 538–539.

The type of locomotive dealt with is particularly suited for use in petroleum refineries, ammunition stores, etc., where care has to be taken owing to danger due to the proximity of explosive gases, etc.

The locomotive is 50 h.p., four-wheel drive, using only one electric traction motor, current being supplied at an infinitely variable voltage. The prime mover is a 12 b.h.p. two-stroke heavy oil engine, in the fly-wheel construction of which is embodied a wheel-operated plate clutch. When this clutch is pulled out and the oil engine stopped, the locomotive is operated from electrical energy supplied from the storage battery.

In order to minimise the heavy losses of electrical energy on starting from rest, and during acceleration, the electro-dynamic features of the well-known "Hopkinson Test" have been used.

The design of this locomotive provides that all operating conditions may be carried out without opening any electrical circuit, and therefore no sparking or burning of contacts occurs. Acceleration is rapid and the standard speed controller gives smooth starting and acceleration.

G. S.

205. The Marine Oil Engine. Prof. C. J. Hawkes. *Petr. Times*, 1928, **9** (472), 181–184.

In reviewing the present development of the Diesel engine in the first Thomas Lowe Gray lecture before the Institution of Mechanical Engineers, Professor Hawkes gave details of the tests carried out by the Marine Oil Engine Trials Committee in the Still and Doxford engines. According to these trials, the most efficient marine engine at the present time was the Still airless-injection two-stroke, the full-load fuel consumption per b.h.p. hour being 0·353 lb., the fuel having a calorific value of about 19,500 B.Th.U. The Doxford airless-injection opposed-piston engine, when tested on shore, had a fuel consumption of 0·416 lb. per b.h.p. hour, the calorific value of the fuel being 18,200 B.Th.U. The average mean indicated pressure of the Doxford engine was 101 lbs./sq. in., and of the Still engine 77 lbs./sq. in. on the combustion side and 7 lbs. on the steam side.

If the indicated thermal efficiency on the combustion side is taken at 43 per cent., and the fuel used has a calorific value of 19,500 B.Th.U., the fuel consumption of the Doxford engine, assuming a mechanical efficiency of 87 per cent., would be about 0·35 lb. per b.h.p., and that of the Still engine, with a mechanical efficiency of 90 per cent., about 0·32 lb. To approach the estimated minimum consumption, an appreciable increase in efficiency in both engines is required.

Trunk pistons are unsuitable for high-speed two-stroke engines and the fitting of crossheads would increase the weight of reciprocating parts and reduce the speed for the same wear and tear factor. The high-speed two-stroke double-acting engine, when developed, was not likely to be less costly to construct when geared to a propellor shaft than with a direct drive, and would be more complicated. The single-acting two-stroke engine is the next step in development, the double-acting two-stroke being the logical line for larger powers, having the advantage of a reduction in the number of cylinders.

The temperature stresses to which liners, covers and pistons are subjected are more important than pressure stresses and the safe continuous rating of an engine depends largely on heat flow.

The improvements in fuel consumption in recent years had been largely due to improved fuel distribution in the combustion zone. Experiments on increasing the turbulence in an airless-injection engine did not vary the fuel consumption, but made the exhaust smoky, and turbulence was not required in the gas engine sense.

Experiments indicated that if the fuel injection was not satisfactory, an improvement in fuel consumption might result from a slight increase in air temperature in the cylinder, but that if injection was good, increased initial compression temperature was accompanied by higher cycle temperatures.

If a two-stroke engine, of low volumetric efficiency when developing its rated power, started readily from cold, the temperature at the end of compression after running for some time was more than sufficient to ignite the injected oil. Better results would be obtained by slightly reducing the compression ratio and warming the jackets to ensure easy starting, and when thoroughly warm to reduce the jacket water temperature and inject the fuel slightly earlier.

Supercharging was not justified, on account of cost, if adopted solely for reducing cycle temperatures, but if higher mean temperatures could be employed without increasing cycle temperatures there was some justification.

G. S.

206. Improvements in or Relating to Temperature-Controlling Devices for Furnace Chambers and Other Apparatus.—N. H. Freeman. E.P. 284,008. Jan. 19, 1928.

The device consists of a tipping mercury switch, which is thrown over by means of an electro-magnet actuated by the temperature controlling device. A pin in the moving end of the hinged armature moved by the electro-magnet engages in a slot on the cradle carrying the switch in such a way that the cradle and the switch with it are given a positive rocking movement when the armature moves. The extent of this movement can be varied by rotation of the member carrying the slot.

P. D.

207. Improvements in or Relating to Liquid Spring Contact Thermometers.—C. Pöhlmann. E.P. 279,853. Jan. 26, 1928.

The improvement consists in the replacement of the mechanical switch used to maintain a constant temperature in the vessel in which the thermometer is used, by a mercury switch. This last can be sealed and so works out of contact with the air. Less trouble is experienced from sparking, and larger currents may be used.

The switch may be either a rotary or tipping switch. The former type may be mounted co-axially with the temperature-recording needle; the latter are worked by a lever arrangement. Switch and levers are all contained in

the case containing the spring. Arrangement is made for setting the instrument so that the current will be cut off at a pre-determined temperature.

P. D.

208. Improvements in or Relating to Liquid Flow Meters.—Shell Mex Ltd. E.P. 284,752. Feb. 1, 1928.

If the delivery pipe placed after a meter is of any great length the meter is apt to give too high a reading as volume delivered, owing to the fact that after the whole of the liquid has passed the meter the delivery pipe empties itself, and in doing so sucks a quantity of air past the meter. This volume of air is registered as liquid.

The simplest means of over-coming this difficulty is to supply a vent pipe placed on the down-stream side of the meter, but it is found that doing this lessens the rate of delivery due to the decrease in suction on the meter exerted by the liquid.

It is proposed to place a specially designed float chamber on the down-stream side of the pump, which is so arranged that the float is lifted by the liquid flowing through the pipe. The lifting of the float closes an air inlet in the top of the chamber by means of a valve. When the flow of liquid stops the float descends and the air inlet valve is opened. At the same time the entrance to the float chamber from the pump is sealed by the float itself, which is made conical in order to ensure correct seating.

P. D.

Anti-Detonation.

209. Comparison of Methods of Measuring Knock Characteristics of Fuels.—G. Edgar. *J. Soc. Automotive Eng.*, 1928, 22, 1, p. 41.

Samples of five different motor fuels were submitted to nine laboratories. The latter were asked to add ethyl-fluid to four of the fuels, in order to make them equal in knock characteristics to the fifth fuel, and also to rate the fuels according to any other system which they ordinarily employed. Three companies employed the bouncing pin method of estimating knock, and agreed in the first series of tests with an error of $\pm$ 10 per cent. Three companies using a method involving the variable throttle opening, with the engine coupled to a dynamometer, agreed in the first series of tests with an error of $\pm$ 40 per cent. Three companies using audibility methods agreed with an error of $\pm$ 60 per cent.

The divergencies between the results are discussed, and are largely atrributed to variations in the air fuel ratio used with the different samples.

W. N. H.

210. The Pressures Produced on Inflammation of Mixtures of (*a*) Carbon Monoxide and Air and (*b*) Hydrogen and Air in a Closed Spherical Vessel.—G. B. Maxwell and R. V. Wheeler. *J.C.S.*, 1928, 15–21.

The paper is a continuation of that published in *J.C.S.*, 1927, 297. A bronze sphere of 4 litres capacity with a highly polished interior was used. Ignition was central; two pressure gauges, one of the Pentavel type, the other of the piston type, were used. The maximum pressures were noted on explosion of varying proportions of carbon monoxide and air, and hydrogen and air. From the observations on carbon monoxide and air the dissociation of carbon dioxide at high temperatures was calculated, the results differ

somewhat from those obtained by Fenning and Tizard and more widely from those obtained by Bjerum.

Similar experiments on the explosion of various mixtures of hydrogen and air give calculated data on the specific heat of steam, as follows :—

Temperature range.	Mean specific heat. Cals. per gram mol.
15–1,400° C.	10·7
15–1,750°	10·6
15–1,950°	10·8
15–2,120°	10·6

W. N. H.

Cracking.

211. The Blumner Cracking Process. *Petr. Times*, 1928, 19, 481.

The method of heating the oil for cracking is claimed to minimise the formation of coke and consists in bubbling the oil under pressure of 35 to 40 atmospheres into an autoclave packed with Raschig rings and filled with molten lead.

The oil is pumped into a cylindrical vessel where it is first heated and then mixed with back-flowing oil, and the mixture is forced at 100° C. into a pre-heater heated by flue gases from the autoclave. The oil is thus heated to 250–300° C., when it is brought into the autoclave at a temperature of 450° C. From here it passes to the decomposers, well insulated vessels, and here the cracking is completed. The cracked oil now passes via an automatic pressure loaded valve into a spraying vessel held at 300° C., where the tar is deposited and flows back, while the light oil goes to an air cooler. The benzine fraction passes to a column and dephlegmator to a condenser where most of it is condensed, but by stripping the gas on active charcoal, 50–100 kilograms per 1,000 cubic metres of oil can be obtained.

From paraffin base oils about 70 per cent. of benzine is obtained, while asphaltic base oils yield 50–60 per cent. of benzine and 24–32 per cent. of asphaltic residue. Brown coal tars, heavy low temperature tar oils and shale oils give 35–50 per cent. of refined benzine and 25–40 per cent. of asphaltic and phenolic residue.

The refined benzine gives 70 per cent. motor spirit and 30 per cent. heavy benzine, resembling petroleum spirit rich in aromatics and unsaturateds and of pronounced anti-knock value.

A. R. S.

212. Cracking Experiments by the Dubbs Process with Distillates from Hanoverian Petroleum.—Dr. R. Heinze, Dresden. *Petr. Zeit.*, Vol. 24, No. 6, p. 237.

As experimental materials for this work the following were utilised :—

(*a*) A gas oil as obtained in a normal distillation process from Hanoverian petroleum.

(*b*) A residue of about 50 per cent. by volume from the same crude material.

(*a*) Cracking of Gas Oil from German Petroleum.

Three experiments were performed with this gas oil, in which the pressure only was varied.

1. *Cracking Experiments at* 14 *kgms./sq. cm. Pressure.*

8,000 c.cms. (specific gravity ·846) = 6768 gms. were distilled under pressure until the residue was still liquid. The pre-heating lasted until the commencement of the distillation, i.e. 2 hours; the pressure-distillation itself took 4¾ hours. The oven temperature rose 2½ hours after the firing to about 620°, was maintianed at 685°–735° during the distillation, and increased towards the end to 795°. The pressure of 14 kgms./sq. cm. was established after 2¼ hours, calculated from the firing, and remained constant to the end of the distillation.

The gravity of every 10 per cent. by volume of distillate from the 8,000 c.cms. oil used was determined. After ½ hour the first 10 per cent. fraction distilled over, and after another 33 minutes the second 10 per cent. fraction, etc. 44·1 per cent. of residual oil was extracted from the oil-containing coke. The pressure distillate (69·7 per cent. by weight) was converted into a fuel with a F.B.P. of 210°. The topped residue from the pressure distillate had the following constants: viscosity 25 4·4° E., flash point 77° C., setting point < 4° C. Small traces were insoluble in benzol. The final yield consisted of 36·2 per cent. fuel, 20·4 per cent. gas oil, 23·6 per cent. residue (heating oil), 1·3 per cent. coke, 18·5 per cent. gas and loss.

2. *Cracking Experiments at* 19 *kgm./sq. cm. Pressure.*

These were carried out under similar conditions as in Experiment 1, except for the pressure. The oven temperature was rather lower, about 670°. The following results were obtained :—

	F.B.P. 210°	F.B.P. 225°
Fuel	29·0% ..	30·9%
Gas oil	16·2% ..	14·4%
Heating oil	35·8%	35·8%
Coke	0·7% ..	0·7%
Gas (0.143 c.b.m.) and loss ..	18·3% ..	18·2%

The gravities of 10 per cent. fractions of the distillate ranged from 0·806 to 0·782. 17·4 per cent. oil was extracted from the oil-containing coke. The pressure-distillate was worked by two methods, either to a fuel with a F.B.P. of 210° or to a fuel with a F.B.P. of 225°. The properties of the oil and fuel produced differed slightly from those of Experiment 1.

3. *Cracking Experiments at* 21 *kgm./sq. cm. Pressure.*

The experimental conditions were the same as in (1) and (2), except for the pressure. The long period of distillation, viz. 5 hours 40 minutes, accounted for the abundant formation of coke; 22·7 per cent. residue was obtained from the coke by extraction, and the pressure-distillate treated as in Experiments 1 and 2. Yields in per centage by weight :—

	F.B.P. 210°	F.B.P. 225°
Fuel	40·2 ..	43·4
Gas oil	14·9 ..	11·6
Heating oil	14·2 ..	14·2
Coke	4·9 ..	4·9
Gas (0·3 c.b.m.) and loss ..	25·8 ..	25·9

The following are the results of distillation on the fuel of F.B.P. 210° in Experiments 1, 2 and 3.

Yields in Percentage by Weight at Different Pressures.

	14 kgms./sq. cm.	19 kgms./sq. cm.	21 kgms./sq. cm.
Fuel	36·2 ..	29·0 ..	40·2
Gas oil	20·4 ..	16·2 ..	14·9
Heating oil	23·6 ..	35·8 ..	14·2
Coke	1·3 ..	0·7 ..	4·9
Gas and loss ..	18·5 ..	18·3 ..	25·8
	100 ..	100 ..	100

There appears from the above table to be no lawful variation. The increase in pressure causes an increase in coke-formation, but no increased yield of fuel. The experiment performed at 14 kgms./sq. cm. pressure appears to be the best.

The fuel produced in Experiment 3 was tested for its anti-knock properties. The toluol value was as follows :—

	%	Individual toluol value.	
Unsaturated hydrocarbons ..	10·5 ..	2·1	Ricardo compression ration, 5·8.
Aromatic „ ..	23·2 ..	23·2	
Naphthenic „ ..	27·2 ..	6·2	
Paraffin „ ..	39·1 ..	—	

Total toluol value, 32·1.

In comparison with experiments with corresponding synthetic mixtures applied in the motor, this fuel corresponds to a mixture of a normal Mid-continent distillate fuel with 21 per cent. benzol.

(*b*) Cracking of Residue from Hanoverian Crude Petroleum.

About 50 per cent. by volume of Hanoverian crude petroleum was distilled by a normal distillation, and the residual 50 per cent. was taken as material for this experiment. The Americans established the following: Specific gravity ·968 I.B.P. 243°, F.B.P. 397°, flash point 252° (Cleveland), burning point 279°, viscosity /100°, 6–7. The gravities of each 10 per cent. by volume distilled ranged from 0·847 to 0·860. Two experiments were performed at 5·25 kgm./sq. cm. pressure, and each 8,000 c.cms. of the initial material (0·968) was submitted to a pressure distillation.

1. Cracking distillation at 5·25 kgms./sq. cm. pressure until the residue was still liquid. The pre-heating period took 2 hours and the distillation lasted 2 hours 40 minutes. The oven temperature was 700–815°. 8,000 c.cms. initial material (0·968) = 7,744 gms. The residue extracted from the coke gave flash point (Cleveland) 99°, burning point i.O.T. 118°, setting point < 0.

Results of Experiment (*b*) 1 *in Percentage by Weight.*

	F.B.P. 210°	F.B.P. 225°
Fuel	27·7 ..	30·7
Gas oil	10·8 ..	7·7
Heating oil	46·0 ..	46·0
Coke	4·1 ..	4·1
Gas and loss	11·4 ..	11·5
	100 ..	100

The fuel produced resembled that obtained in experiment (*a*).

	%	Individual toluol value %	
Unsaturated hydrocarbons ..	15	3	Ricardo compression ratio, 5·8.
Aromatic ,, ..	25·6	25·6	
Naphthenic ,, ..	9·5	2·4	
Paraffin ,, ..	49·9	—	

Total toluol value, 31·0.

82·4 per cent. of residual oil was obtained from the coke by extraction. The pressure-distillate was treated as before.

2. Cracking distillation at 5·25 kgms./sq. cm. pressure carried out to dryness. 8,000 c.cms. initial material = 7,744 gms.

Fifty per cent. oil was extracted from the coke.

Results of Experiment (*b*) 2 *in Percentage by Weight.*

	F.B.P. 210°	F.B.P. 225°
Fuel	30·8	33
Gas oil	34·7	32·5
Coke	21·7	21·7
Gas and loss	12·8	12·8
	100	100

	%	Individual toluol value.	
Unsaturated hydrocarbons ..	12·3	2·5	Ricardo compression ratio, 5·8.
Aromatic ,, ..	26·6	26·6	
Naphthenic ,, ..	9·9	2·5	
Paraffin ,, ..	51·2	—	

Total toluol value, 31·6.

The fuel resembled that of (*a*).

In order to produce a valuable fuel the pressure-distillate from the cracking experiments (*a*) and (*b*) were refined by treatment with plumbite-lye and then subjected to a steam distillation.

W. S. C.

213. Improvements Relating to Processes for Producing Light Hydrocarbons. E. S. Andrews. E.P. 285,199. Feb. 16, 1928.

The heating system of this process consists of two furnaces, within which heating tubes are situated. The tubes are arranged in five horizontal coils connected to each other in a certain manner. Two feed tanks receive the oil to be cracked from a convenient source, and these tanks have a water connection at the bottom. The water-pipe is also connected to the bottom coil in the first furnace. The oil is forced into the top coil of the first furnace by the water pressure on the feed tanks and passes through the first coil. The water coil is connected to the end of the first oil tube and the water and oil mix before proceeding into the other three coils, which are connected in series. The mixture passes thence into the second furnace, where the tubes are connected in series, flowing downward. The cracked vapours are then discharged into a large vertical cylindrical cooler, which is fitted with perforated baffle plates in the middle part, whilst flat circular coils occupy the bottom and top parts. These coils serve to regulate the temperature of the cooler, either water or steam being admitted as required, to cool or heat the vapours in order to obtain the desired product. Three of these coolers are connected in series and receiving tanks are placed convenient to each.

By controlling the oil and water feed any desired temperature may be maintained in the vapours issuing from the cracking coils. The pressures used are approximately atmospheric. The process may be run continuously, as residues are continuously recycled and the deposition of carbon practically eliminated. The cracking coils may be made of an alloy of iron and silicon, 3 per cent. to 10 per cent. of the latter, for it is found that the silicon apparently acts as a catalyst and improves the quality of the spirit.

R. P.

214. Process for Making Gasoline. O. Behimer. U.S.P. Re. 16,887. Re-issued Feb. 14, 1928. Original appl. filed Nov. 21, 1918. Divided April 6, 1923 and again divided August 13, 1925.

In prior methods of cracking oils, difficulty has been experienced on account of carbon formation occurring on the tubes or stills exposed to the heat necessary to carry on the operation. In the novel process disclosed, substantially all the cracking occurs on a vessel to which no external heat is applied, except as necessary to compensate for heat losses, the oil prior to its introduction to such vessel having been subjected to a high degree of cracking heat, and the excess heat of the oil itself is used to effect its decomposition. The removal of the cracking operation from the heating zone reduces the danger usually present in processes where cracking is carried out in highly heated stills exposed to direct heat.

In this process a cyclic system is employed in which a circuit of oil is constantly maintained under varying liquid and vapour phases. The oil, on entering is subjected to a high degree of heat, but the time factor is so controlled that there is little deposition of carbon, as the oil is rapidly removed from the heating zone and conducted to the cracking zone where it undergoes cracking without being subjected to additional heat. Oil cracking is not dependent only on pressure and temperature, but also on the time necessary for the decomposition to take place.

In this process the time element is controlled by regulating the rate of heating in the heating zone so that the oil reaches the desired cracking temperature at about the time the oil emerges from the heating coil and before any substantial deposition of carbon takes place. Subsequently the highly heated oil in an incipient state of decomposition is delivered into the cracking zone, where conditions are maintained to complete the cracking operation. The bulk of the carbon formed is removed from the cracking zone with portions of the residual oil. The cracked vapours are passed through a dephlegmator and reflux oil is recharged while the spirit vapours are condensed and removed from the system.

The apparatus consists of a tubular heating coil contained in a furnace and an enlarged drum which is suitably insulated to prevent loss of heat. It is equipped with a draw-off pipe and vapour outlet, which extends to a separator and terminates in a condenser for light spirit.

R. P.

215. Art of Cracking Hydrocarbons. J. E. Bell. U.S.P. 1,660,856, Feb. 28, 1928.

This invention is another method for the prevention of carbon formation in pressure still cracking plant. The apparatus comprises a horizontal still which is divided into compartments by baffle walls reaching above the oil level. The vapour space is common and connects to a dephlegmator, which is placed directly above the first compartment. The still itself is not heated, but from its bottom, coils of tubing depend into a heating chamber. Each compartment of the still has a separate coil and the heating chamber is divided

into sections, each containing one of the heating coils. The flue gases pass successively through the sections of the heating chamber, becoming cooler as they progress through the series. The oil in the still passes successively through the compartments in parallel flow to the flue gases so that it is gradually subjected to milder conditions of cracking as the oil concentrates. The coils within the heating chamber also gradually increase in size in order to give a greater heat absorbing surface to compensate for the fall in temperature of the flue gases.

The flue gases are also recirculated, part of the cooler gases being returned and mixed with the gases issuing from the furnace in order to control the process and at the same time save fuel.

By this method the heating surfaces most apt to suffer are protected as the fresh oil mixed with reflux from the dephlegmator is subjected to the most severe conditions, these being least likely to deposit carbon, whilst the more concentrated and partially cracked oil, which contains more tar, is subjected to milder conditions of cracking to prevent coke deposits on the metal surfaces. The method of heating in coils depending from the still bottom also causes a positive circulation which has the effect of maintaining the oil in the different compartments at a constant temperature.

R. P.

216. Apparatus for Treating Hydrocarbons. G. Egloff and H. P. Benner. U.S.P. 1,659,397, 1928. Appl. Sept. 2, 1926.

The apparatus consists of a still having a stationary portion and a rotating portion and fitted with a heating element extending through the still. The heating element is capable of being rotated, preferably in the opposite direction to the rotation of the still. Cracking stock is introduced into, and residues and vapours removed from, the stationary part of the still.

H. G. S.

217. Improvements in or Relating to Apparatus for Cracking Carbonaceous Materials. A. E. Bianchi. E.P. 277,404, Sept. 8, 1927.

The apparatus is designed for the cracking, catalysing and hydrogenation o. carbonaceous materials, such as oils, tar, naphtha, schist or ground coalf It consists of a series of troughs with revolving agitators. The troughs are provided with chains depending from reticulated screens and are capable of being vibrated by the revolving agitators. The chains prevent dust from passing off with the vapours and a permanent cloud of fine coal-dust is formed in the space above the level of the carbonaceous material. This dust is kept in continual contact with the vapours and begins the catalytic action, after which the vapours come into contact with catalysing substances coated on the upper parts of the apparatus or on the reticulated screens or chains and a further pyrogenetic decomposition or transformation is obtained, while the hydrocarbons are still in the form of gas.

R. P.

218. Process of Cracking Oils. J. C. Clancy. U.S.P. 1,658,116, Feb. 7, 1928.

In catalytic processes of cracking and hydrogenation of oils for the production of gasoline, difficulties are usually encountered in the form of carbon and sulphur, which coat the catalyst and render it useless very quickly. In the present invention these difficulties are overcome by acting on the oil with a molten body containing a cyanogen compound, in presence of a solid catalyst.

The reaction chamber consists of a vertical cylindrical chamber heated externally and packed with the catalyst which preferably is composed of copper in the lower part, cobalt and copper in the middle and nickel in the top sections. These metals are in the form of Rashig rings of about 1 inch in diameter. The reaction chamber is sealed top and bottom by the fluids entering or discharging. Oil and hydrogen or hydrogen containing gases are entered near the bottom of the tower and a molten mixture of 25 parts sodium thiocyanate and 75 parts sodium cyanide enters at the top of the tower and travels downward, counter-current to the flow of hot gases and vapours. Thiocyanates are formed by the action of the cyanide on the sulphur compounds contained in the oil and are carried out of the system, thus keeping the catalytic surfaces clean and active. The carbon and unvaporised portion of the oil are also carried out with the molten cyanides.

R. P.

219. Process for Treating Petroleum Hydrocarbons. W. M. Cross. U.S.P. 1,643,446, Sept. 27, 1927.

The invention is similar to previously patented Cross processes and consists of the same plant, but differs in so far that, instead of introducing a blending oil into transfer line or evaporating tower, a jet of superheated steam is injected between the reaction chamber and evaporating tower for the purpose of assisting the distillation of the oil in the evaporator and driving off from the oil body a sweeter product than would result from distillation due solely from the heat contained in the oil. Superheated steam is used for this purpose. It is also claimed that the residue from the evaporator is of better quality when steam is used.

R. P.

220. Process for Cracking Hydrocarbon Oils. C. P. Dubbs. E.P. 284,507, 1928.

The invention is particularly directed to that type of cracking process (*e.g.*, E.P. 249,604) in which the raw stock is heated under pressure to cracking temperature and then passed to an expansion or reaction chamber, the insufficiently cracked products being withdrawn for further treatment and the cracked gasoline condensed. In this invention incondensible gases, produced in the process or introduced from an internal source, are heated to a temperature substantially higher than that of the oil in the transfer line and passed in regulated proportions into the reaction chamber.

H. G. S.

221. Conversion of High Boiling into Low Boiling Hydrocarbons. L. Edeleanu, Assr. to Allgemeine Gesellschaft fur Chemische Industrie, Berlin. U.S.P. 1,661,565, March 6, 1928.

The process comprises a treatment of petroleum distillates prior to their further fractional distillation by cracking processes. The inherent disadvantages of all cracking processes are that asphaltum and bodies rich in carbon, always present in cracking stock, produce during the cracking operation a large amount of coke, resulting in frequent shutting-down of the still for cleaning. Also, nitrogen and sulphur compounds often present lead to corrosion of equipment. These drawbacks are minimised according to this invention by treating the cracking stock prior to cracking with liquid sulphur dioxide, which is a good solvent for the above bodies. Methods of treatment of petroleum oils with liquid sulphur dioxide for the removal of substances soluble therein are described in U.S.P. Nos. 911,553 and 1,526,665. The following example is given: By treatment with liquid sulphur dioxide of a

Texas Luling gas oil having a specific gravity of 0·889 and a sulphur content of 0·8 per cent., a gas oil with a specific gravity of 0·865 and sulphur content 0·24 per cent. is obtained. Both oils were cracked separately under equal conditions of pressure (85 lbs.) and temperature (77° F.) and gave the following products :—

	Non-treated gas oil. Per cent.	Treated gas oil. Per cent.
Cracked distillate	82 ..	90·5
Gasoline	45 ..	53·7
Coke	4·6 ..	1·5
Sulphur in cracked distillate	0·34 ..	0·09
Sulphur in gasoline	0·1 ..	0·06

In the non-treated gas oil remaining as a residue of the process a sediment of about 6 per cent. of carbon and asphaltic matter forms, while in the treated gas oil there will appear only traces of sediment. If the cracking of gas oil is affected by pressure and catalytic action, the advantages of such preliminary treatment with liquid sulphur dioxide are more striking.

H. S. G.

222. Improvements in the Decomposition of Gaseous or Vaporous Hydrocarbons by Means of Water Vapour.—I. G. Farbenindustrie Aktiengesellschaft. E.P. 265,989, Sept. 22, 1927.

It has been found that the process of producing hydrogen and carbon monoxide and pure hydrogen from aliphatic, aromatic and hydroaromatic hydrocarbons may be carried out by cooling the highly heated, decomposed gases with water, whereby the water is heated and large amounts of water vapour condensed from the hot gases.

The resulting hot water is brought into contact with fresh hydrocarbon gas to be cracked, thus warming and charging them with aqueous vapour. This mixture is introduced into the reaction chamber. If the process is carried out in presence of oxygen or gases containing oxygen, these may be pre-heated and charged with water vapour in the same way.

If pure hydrogen is to be produced, the gas from the decomposing chamber is passed over a catalyst, such as activated iron chromium catalyst, adapted to effect the converison of carbon monoxide and water into hydrogen and carbon dioxide. The carbon dioxide may be easily eliminated and pure hydrogen obtained.

R. P.

223. Improvements in the Manufacture and Production of Valuable Liquid and Other Hydrocarbons and Derivatives thereof by the Destructive Hydrogenation of Tars, Mineral Oils, Resins, Asphalts and Like Initial Materials.—I. G. Farbenind. Akt.-Ges. E.P. 282,814, 1926.

Modification of E.P. 256,964 (see abs. 256) applied to raw materials, which are liquid or fluid under the conditions of working. From 5 to 10 per cent. of brown coal added to the tar, etc., is sufficient to prevent coking, though quantities up to 30 per cent. may be used. Catalysts, either added to the charge or arranged in the reaction vessel may also be employed. Hot parts of the apparatus are preferably made of materials which are stable under the working conditions, e.g. iron coated with chromium or alloys rich in chromium, such as chromium-nickel-steel substantially free from carbon.

H. G. S.

224. Process and Apparatus for Cracking Oils.—W. F. Farragher and W. A. Gruse. U.S.P. 1,640,444, August 30, 1927.

This invention provides an apparatus designed to prevent the formation of carbon and its objectionable effects during the cracking operation. The "air lift" principle is utilised to induce a rapid and positive circulation of the oil in contact with the heated surface. Troublesome mechanical stirrers or agitating devices which have been tried for the same purpose are thus eliminated, and the plant can be more easily made gas-tight.

The plant consists of an upright heating tube having an oil inlet. A suitable heating furnace surrounds the middle part of the tube and the upper part has a dephlegmator, whilst the lower part (not heated) is a tar chamber, from which heavy residue may be drawn off. Within the heating tube is a centrally supported eduction tube with a gas inlet pipe arranged so that it discharges fixed gas, obtained in the process, upwardly. This discharge of gas, rising through the central tube, causes a circulation of the oil upwards in the central tube and downwards through the annular space in the still.

The entire body of the oil is thus heated uniformly and can be maintained at a constant cracking temperature. The gas introduced escapes along with the vapours of the cracked oil. These are separated and the gas is returned into the system whilst the cracked product passes out to receivers.

Various modifications of the apparatus are described, as the method can be applied in the reverse direction, i.e. by causing the gases to pass up the annular space. The method may also be used in conjunction with a boiler-shaped still, by having depending legs which are provided with the "air-lift" and exposed to the furnace heat. In this case the still acts as a reservoir, from which the oil circulates.

R. P.

225. Process of Transferring Gases and Vapours.—G. E. Fox, Assr. to Standard Oil Development Co. U.S.P. 1,659,948, Feb. 21, 1928.

Oil to be cracked is fed into a coil maintained at a temperature of about 700° F. under a pressure of about 25 lbs. per square inch. The rate of flow through the coil is sufficiently rapid to vaporize a substantial proportion of the oil in the coil without cracking and coke formation therein. The oil passes from the coil into an injection device, where gas under high pressure, preferably about 1,000 lbs. per square inch, is supplied from a compressor. The low pressure vapours and liquids are carried through a constricted passage into a drum in which a pressure of about 350 lbs. is produced, thus greatly increasing the pressure on the vapours without the use of a pump. The high pressure gas is preferably heated to about 900° F. In the drum, conversion of the oil takes place and vaporous and liquid products are taken off to a bubble tower operated at 60 lbs. pressure or less, the pressure being controlled by a suitable valve. The vapours are taken off from the upper portion of the tower for condensation and the residuum is drawn off from the bottom, as required. Gas injection in the manner described provides a reliable and economical method for producing the desired pressure in a digesting chamber operated in connection with a vapour phase or semi-vapour phase heating coil without the use of any mechanical pumps.

H. S. G.

226. Process and Device for Converting Heavy Hydrocarbons into Light and Stable Hydrocarbons.—P. Grené. E.P. 285,668, 1928.

The oil or tar to be converted is passed in the vapour phase, together with suitable neutral gases, through tubes, each of which is packed, in succession, with (*a*) a non-metallic catalyst, e.g. coke, activated carbon, etc., (*b*) oxide

of manganese, (*c*) a second layer of the non-metallic catalyst, (*d*) a hydrogenating catalyst, such as nickel.

H. G. S.

227. Process and Apparatus for Cracking Oil.—E. C. Herthel. U.S.P. 1,660,647, Feb. 28, 1928.

The process is the production of gasoline from heavy oil, such as gas oil, and is designed to minimise the danger of carbon deposition on heating surfaces, such as are encountered in pressure stills.

The heating gases are circulated first over an auxiliary heating coil and are then admixed with hot gases at a lower temperature before being applied to the still. The auxiliary heating coil is arranged to discharge into the still which is provided with reflux towers. The reflux either with or without fresh oil is recycled through the auxiliary coil, thus providing an oil which is not liable to deposit carbon in the tubes. Tar-laden oil may be withdrawn from the still during the progress of cracking to prevent carbon deposition and the rate of withdrawal and rate of supply of fresh oil may be regulated to maintain the tar content of the still charge below saturation point.

The tempertaure and pressure employed can be varied with different charging oils. For ordinary gas, oil pressures of from 90 to 125 lbs. may be used, but with ligher oils 300 to 325 lbs. may be necessary.

R. P.

228. Art of Cracking Hydrocarbons.—E. W. Isom. U.S.P. 1,661,073, Feb. 28, 1928.

In this process the oil is passed in one cycle through a tubular cracking still and discharged into a shell still from which the vapours are taken off. The cracking coil is contained in a heating chamber wherein hot gases from the fire-box are circulated, after being mixed with a part of the gases which have passed over the tubes and are returned to dilute and temper the furnace gases. The vapours from the still pass into a dephlegmator and through a condenser. Reflux oil from the dephlegmator may be returned with fresh oil. The unvaporised residue discharged into the shell still may be withdrawn from the system or additional fresh oil, particularly oil of a heavier character than that passing through the pipe still, may be supplied to the shell still and subjected to a cracking operation by the heat of the products therein.

The shell still may be heated by a separate furnace.

By re-circulation of the flue gases fuel economy is improved, high combustion temperature may be used without detriment to the operation and without subjecting the equipment to severe strain. Re-circulation of the flue gases increases the volume and hence the velocity, and allows a greater amount of heat to be transferred to the oil in a shorter length of travel.

R. P.

229. Oil Cracking Process and Apparatus.—W. C. Kirkpatrick. U.S.P. 1,646,543, Oct. 25, 1927.

In the majority of cracking processes carbon is formed in such quantity or in such a physical condition as to make it difficult to discharge and usually necessitates periodical shut-down of the still for cleaning. It is the object of the invention to provide a process which yields a finely divided and dry carbonaceous deposit and to provide a means for removal of this deposit while the plant is in operation, whereby the apparatus will run continuously without interruption for cleaning.

The plant has a furnace and heating chamber; the latter contains an upright cylindrical cracking still. The heating chamber is divided by a vertical

partition wall so placed that the hot gases from the furnace pass down one half of the still and up the other half. The still also has a partition wall, in its interior, which extends from the top to within a short distance from the bottom. The oil is injected into the cooler half of the still, travels downwards, under the internal partition and upwards through the increasingly hot half of the still, thence to the outlet. The lower end of the still is cone-shaped and has a carbon outlet leading to two carbon pots which have valve-controlled outlets. These carbon pots may be used alternatively, one being filled while the other is discharging.

R. P.

230. Improvements in or Relating to the Conversion of Hydrocarbon Oils.—F. Lamplough. E.P. 277,419, Sept. 15, 1927.

Petroleum is brought into intimate contact with coal and the mixture heated with or without the application of pressure. It is claimed that the petroleum is hydrogenated and the light fractions produced are, in appearance and density, equivalent to the best quality of motor spirit.

The coal is intimately mixed with the heavy oil in so finely-divided a condition as to produce a stable dispersion of coal in oil, and this dispersion is subjected to a heat treatment. After heat treatment the liquid product is preferably maintained under pressure in a vessel so that solid matter may be settled prior to distillation. Such distillation may be continuous and fractional condensation may be effected.

The proportion of coal to oil may be from 10 per cent. to 50 per cent. and the coal is preferably prepared by the froth flotation method to free it as far as possible from ash.

The suspension of coal in oil is pumped through a heating coil at about 400–500° C. and at a pressure of 300–600 lbs. per square inch.

Thence the mixture passes into a settling chamber where the ash settles to the bottom and the oil may be decanted. The duration of the treatment may be from 30 to 60 minutes. After settling, the pressure is released and the light oil is distilled off, leaving a sludgy residue which may be treated with superheated steam to cause further vaporisation. The final residue is removed through a manhole door in the bottom of the chamber.

R. P.

231. Refining Petroleum Oils. P. McMichael. U.S.P. 1,658,171. Feb. 7, 1928.

The patent describes a method of refining cracked distillates in which treatment is effected by means of an alkaline aqueous solution of sodium chromate having a gravity of not less than 20° Bé. After this treatment the cracked oil is washed with dilute sulphuric acid. The sodium chromate washing may be preceded by treatment with either an aqueous solution of caustic soda or with dilute sulphuric acid.

W. H. T.

232. Apparatus for Cracking Oil. R. S. Pershing. U.S.P. 1,661,196. March 6, 1928.

An apparatus is described for cracking oil for the production of gasoline without the use of high pressures. It consists of a cylindrical horizontally arranged still casing, suitably mounted, and is provided with dome and vapour line leading to a condenser. A gauge tube is provided at one end and a manhole at the other. Extending longitudinally and concentrically within the still is a heater embodying a cylindrical tube of copper closed at one end. The heater is supported so that it can be moved longitudinally. Inside the

heater are spacers of insulating material with longitudinal openings for copper conductor rods by which the heat is provided. Arranged above and parallel to the heater are oil supply pipes provided with sets of nozzles converging downwardly through which oil is sprayed upon the upper portion of the heater. In practice the still is maintained about one-third full and the heater operated at a temperature varying from 212° F. or below to 1,500° F. The pump is started and oil passes through the supply pipe and is sprayed upon the heater. Gasoline bearing vapour passes up while the liquid gravitates from the heater to the lower portion of the still, from which it is withdrawn through valves in such amount that the liquid level in the still remains constant. The oil so withdrawn passes to the intake end of the pump which is also connected with a fresh oil supply pipe.

H. S. G.

233. Cracking Still. A. A. Phelan. U.S.P. 1,646,929, Oct. 25, 1927.

The object of the invention is to remove the free carbon formed in the cracking process in a continuous manner, thus lessening the wear and tear of the apparatus, lengthening its endurance and promoting its efficiency. By the continuous removal of the carbon as it forms, local overheating of parts of the apparatus is prevented and blistering, buckling or sagging of overheated metal is obviated.

The plant consists of a vertical cylindrical still, having at the top a heated oil inlet and a vapour outlet. A vertical partition extends from the top, between the inlet and vapour outlet, to the bottom of the still, thus dividing it into two chambers. There is an opening at the bottom of the partition affording communication between the chambers. In the liquid receiving chamber there are a series of baffle plates extending from the partition at an angle downwardly, and immediately beneath each baffle plate there is an opening in the partition for vapours to pass into the vapour chamber.

The lower part of the vapour chamber is divided into two parts: the one adjacent to the partition being a carbon precipitation chamber, and the other an overflow compartment by means of a short vertical wall extending from the bottom of the still. From this wall two baffle plates extend into the precipitation chamber at an angle downwardly, and two similar baffles extend from the partition, thus causing the liquid to travel upward in a zig-zag path to overflow into the overflow compartment. From the latter chamber the oil passes out through a discharge valve. The carbon precipitation chamber is supplied with an outlet pipe and valve for the removal of carbon and heavy oil.

R. P.

234. Heating and Cracking Oils. E.P. 283,032. Power Specialty Co., New York. April 20, 1927.

An improved method is described of heating oil to a cracking temperature with little or no cracking, and simultaneously superheating steam to a high temperature. Oil is passed at a relatively high velocity through tubes of a tubular oil heater located in the high-temperature portion and operating so that the oil-heating tubes will not cool the heating gases below about 1,500° F. The available heat in the heating gases is utilized in superheating steam to a high temperature, the combination of oil heating and steam superheating operating with high thermal efficiency.

The heater comprises a combustion chamber and a tube containing heating chamber, separated by a bridge wall over which the heating gases pass. The oil-heating section comprises a bank of horizontally disposed tubes surrounded by externally corrugated cast-iron casing sections and located in the

upper portion of the chamber, and a row of tubes lining the roof of both chambers extending transversely to the length of the bridge wall with the portions directly above the combustion chamber exposed at their undersides; while their upper sides are received in channels in the brickwork roof, the other portions are encased in cast metal blocks. The oil to be heated passes through an inert connection to one of the tubes in the bottom row, and then back and forth, first through the tubes in the same row, then similarly through the tubes in the next row above, and so on, through a connection to the adjacent end of one of the roof tubes, and serially through these tubes to the heater oil outlet.

The superheater tubes are similar to the well-known Foster superheater tubes in that, like the tube elements, each tube is surrounded by tubular externally corrugated cast-iron casing sections. The steam is supplied through an inlet connection to a superheater inlet header to which one end of each tube in the lower adjacent row is connected. Adjacent tubes in the adjacent rows at the different levels are connected at one side of the heater by return bends and at the opposite side of the heater by headers. The steam, after passing through the tubes at the different levels, is withdrawn through the superheater outlet header to which one end of each tube in the upper row of such elements is connected. Excessively high superheat temperature may be avoided by injecting a spray of water into the superheater inlet header. Available heat in the heating gases, passing away from the superheater, may be recovered by any suitable waste heat recovery means, and is preferably utilised in preheating the oil passing to the oil inlet of the tube system.

H. S. G.

235. Improvements in Cracking Hydrocarbon Oils or their Distillates by Distillation under Pressure. S. Stransky. E.P. 267,958. March 1, 1928.

The process consists of distilling the oil under pressure at a temperature of about 400° C. This temperature is maintained constant and equilibrium between the cracked and uncracked oil is permanently disturbed by removal of the light fractions very quickly by introducing large quantities of heat per unit time. This is carried out by use of electric heating resistances of large heating surface distributed throughout the space within the still, combined with an agitating device whereby the temperature difference between the oil and the heaters is reduced to a minimum.

The ratio of the volume of liquid in the still to the gas space is maintained constant at that particular ratio most favourable for the material under treatment, found by experiment at the beginning of the operation. These conditions are secured by means of automatically working controls. The process may be worked continuously by means of stills arranged in series. It is claimed that from Galician gas oil outputs of 60. per cent. with boiling-points between 35° and 170° C. have been obtained. Residues amounted to about 10 per cent., and the formation of gas was reduced to a minimum and seldom exceeds 8 per cent. of the weight of the original material. R. P.

236. Combined Distilling and Cracking Process and Apparatus Therefor. M. J. Trumble. U.S.P. 1,659,930. February 21, 1928.

The apparatus is intended for the production of gasoline from solid material such as shale, coal or lignite. The plant consists of a retort, a cracking still, a primary and a secondary condenser, with the necessary receivers and pumps.

The retort is provided with a steam pipe for the admission of steam which is used as the heating medium. Vapours are taken off at the top of the

retort, and pass, at a pressure of about 150 lbs., to a steam turbine engine or the like, which is directly connected to an electric generator. The expansive force of the vapours produce power which may be utilised as required. The vapours from the exhaust side of the prime mover are delivered into the primary condenser, and uncondensed vapour passes them into the secondary condenser, where they are recovered and sent to storage. The condensate from the primary cooler is pumped into the cracking still and the cracked vapours are led into the vapour pipe leading from the retort to the prime mover, where they are utilised. By combining the vapours from the retort and the still, condensation of tars and objectionable substances in the working parts of the prime mover is avoided.

R. P.

237. Process for Destructive Distillation of Hydrocarbons.. C. W. Turner. U.S. P. 1,647,026. October 25, 1927.

The process consists of passing a mixture of steam and oil through a pair of vertical chambers, heating the mixture, conducting the vapours into another pair of similar containers wherein the vapours are heated to a high temperature and then expanding and fractionating the vapours. The plant consists of a water and two oil feed tanks. The first two vertical heating chambers are contained in a furnace which also contains a coil situated in the roof for vaporising the water. The steam and oil are injected into the first chamber near the bottom and the mixture vaporises from the top of this vaporiser. A tube leads to the bottom of the second chamber or vapour heater. Means are provided for regulating the proportions of oil and water entering the plant at a pressure of about 100 lbs. per sq. in. After the vapours have been preheated they pass through the second pair of vertical chambers wherein the temperature is raised to within the range 425° to 875° C. and in the second to within the range 660° C. to 1100° C.

From the last heater the oil goes into an expansion chamber then successively in three vertical tanks wherein the oil cools and deposits heavy oils. The vapour temperature has now dropped to about 500°C. The vapour proceeds into a scrubber near the bottom. This tank is maintained half full of water and the vapours must pass through this into the upper part. In doing this the vapours lose part of their heat, and all impurities in the form of carbon and certain of the more solid hydrocarbons and substances which would otherwise discolour the distillate, form a layer on the top of the water. Means are provided for drawing off this layer. Thereafter the vapours travel through a series of ten vessels wherein different fractions are condensed, and finally through three gasoline condensing tanks.

R. P.

See also Abstracts Nos. 163, 165, 167, 170, 172, 177 and 246.

Oil Field Technology.

238. Late Developments in Electric Methods of Locating Depth of Water Intrusion in Oil Wells. J. H. Tinkham. *Oil Age*, 1928, 25 (3), 27–28.

In order to successfully shut off water it is necessary to know the exact point of entrance, and during the last two years an electrical method for

this has been developed. The principle involved is the measurement of electrical potential difference between two dissimilar metals moving through an electrolyte varying in concentration and in chemical characteristics. In effect the well is used as a large wet cell and the voltage measured at all depths by noting the potential difference between two dissimilar elements lowered from top to bottom.

The equipment consists of a reel of insulated twin conductor cable, to which is connectedan apparatus containing the two metals and a millivoltmeter to record change in potential difference. An automatic depth measuring device is provided.

When the fluid in the well is at its normalhy hydrostatic head, the potential curve from the normal well fluid is recorded. A suitable electrolyte is then evenly distributed by a special boiler throughout the entire fluid zone. The recording device is then passed through and readings taken to ensure even distribution of the electrolyte. The fluid level is then lowered by removing some of the upper contents, and the formation water, seeking its normal level, flows from its source and changes the chemical characteristics of the fluid at its point of entry. The instrument is again passed through the fluid and an abrupt change in the millivoltmeter reading noted where the chemical contents are changed.

As an additional check fresh water is run in at the top of the hole, forcing the electrolyte solution out into the formation at the point of entry of formation water. As the point of contact of fresh water and solution is thus lowered, the depth of contact is recorded by millivoltmeter readings, and when the lowest point of water entry is reached the readings remain constant.

The results are recorded as curves on a chart and clearly indicate the depth at which water is entering. G. S.

239. Cementing Bottom Hole Water and Proper use of Accelerators. Vietti and Oberlin. *Oil and Gas J.*, Feb. 16, 1928.

The shutting off of bottom hole water in oil wells is an accepted practice in the industry, lead wool, cement and limit plugs being used for this purpose. Before cementing a hole it must be carefully prepared by freeing the surface of sand and mud, cavings should also be filled in. Since the setting of the cement is destroyed by agitation, the hole should be loaded with oil or oil and water and allowed to stand till pressures become equalized.

High grade Portland cement mixed with 2 per cent. of calcium chloride to accelerate setting and the cement mixture should be made with the water in which the cement is to set. The cement should be placed with as little admixture of well water as possible. A. R. S.

See also Abstracts, Nos. 146 and 193.

Petroleum Geology.

240. Seminole Area Sub-surface Formations. J. L. Dwyer. *Oil and Gas J.* 26, 37, p. 70.

A chart is given below representing the seminole sub-surface conditions; the section is read from above downwards to the oldest rocks, these comprehending the most interesting parts of Seminole well logs.

GENERALIZED SECTION, PRE-PENNSYLVANIAN STRATIGRAPHY, MAJOR SEMINOLE AREA.

Formation.	Beds.	Description.
Mississippian	1. Lower Caney and Mayes beds. 150′–450′ 40′–400′	Dark brown shale. Granular calcareous grit.
	2. Sycamore lime, 10′ ..	Green, glauconitic shale and lime.
	3. Woodford (Chattanooga), 10′–140′	Dense brown to black purple shale. Cherty at top.
	4. Misener sand, 0′–15′ ..	Irregular, round-frosted subangular sandstone.
Devonian ..	5. Hunton lime, 0′–150′ ..	White to pink crystalline limestone.
Silurian ..	6. Sylvan shale, 15′–110′ ..	Greenish gray to gray fine grained pyritic shale, often darker near base.
	7. Viola lime, 20′–70′ ..	Coarsely crystalline—white, gray and pink.
Ordovician	8. Simpson lithographic dolomitic limestone, 0′–75′	Dense, buff to brown lime, imperfectly dolomitised. Very sugary texture towards base.
	9. 1st Wilcox sand, 70′–125′	Irregular sized, round-frosted and subangular sand, limy.
	10. 2nd dolomite and green shale, 0′–40′	Brown sugary lime may be sandy, and dark green shale.
	11. 2nd Wilcox sand ..	Even grained fine subangular sand.
	12. Simpson sand and green shale.	Uneven grained round-frosted sand and dark green shale, not limy.

CHARACTERISTICS.

1. When the "Hunton Lime" is present a full section of "Sylvan shale" follows.

2. Wells showing shale in the "Lower Caney formation" have a full section of the "Mayes stratum."

3. Wells showing the "Sycamore lime" may expect a full section of the "Woodford section."

4. The Pennsylvanian formations occupy a position slightly above the "Caney," and generally run to a depth of about 3700 ft. below the surface at Seminole. The "lower Caney" is normally reached at a depth of from 3750′ to 3800′.

5. There is a gradual thickening of formations from north to south corresponding with approach to the Arbuckle Mountains, one of the sources of supply of the sediments.

6. The "*first Wilcox sand*" was the principal oil producing sand in the old Seminole City pool.

7. *The* "*Hunton lime*" was the first formation to yield commercial oil production, and caused the drilling activity which resulted in the discovery and exploration of the "Wilcox sand."

8. *The "Viola lime"* is of unusual importance because it is the formation most used in making calculations upon the position of the "Wilcox." Most structural maps have minus datums on the "Viola lime."

J. E. M. H.

241. THE GEOLOGY OF THE SEMINOLE FIELD. *Petr. Zeit.*, 24,4 1928.

The opening up of the Seminole field had a very great effect upon the oil market last year. The falling off in the oil production of the N.S.G. suddenly gave place to over-production and world prices fell sensibly. The following notes taken from S. Power's paper in the *Bulletin* of the American Association of Petroleum Geologists (October, 1927) will be of interest.

North of the Arbuckle Mountains in Oklahoma occurs the Seminole uplift with the oilfields of Seminole, Searight, Bowlegs and Little River—this area was formerly considered unfavourable for oil production.

On March 7, 1926, the Seminole field was discovered from a boring made by the "Indian Territory Illuminating Oil Company."

This yields now daily 1100 barrels from the Silurian Hunton limestone. The depth was 4012 ft. On July 6, 1926, another company obtained 60 barrels daily from the "Wilcox sand," stratigraphically lying beneath the above-named. Next came the "Garland Independent Company," July 16, 1926, which obtained 1500 barrels daily at 4069 ft. and later on 8000 barrels at a greater depth; later on other discoveries were made (Wilcox sand).

The upper carboniferous in the Seminole field outcrops and extends to a depth of 3700 ft. downwards—its lowest horizons being absent. Beneath came the lower carboniferous ("Mississippian"). Next follows the "Hunton limestone" (Silurian). This in certain places is 30 ft. to 90 ft. thick, but in others attains 350 ft. Where this limestone is absent oil bearing sands may occur. Beneath all these occurs the Ordovician—with 60 ft. or 100 ft. of "Sylvan" shales, beneath which occur the thicker "Viola" limestone and the "Simpson Formation," containing the four beds of "Wilcox sands" between 20 ft. and 80 ft. thick where oil bearing. Much dolomite occurs some 15ft. to 60 ft. above the first oil sand. Beneath the second of these, green shales occur.

To the north of the Arbuckle Mountains and west of the Ozark uplift the shales have a slight westerly dip of 30 ft. to 100 ft. per mile—near the Seminole uplift the dip is 90 ft. per mile and fissuring and undulations occur above. East and west there are no discolorations or undulations. The shales beneath the upper carboniferous are acutally traversed by faults (1500 ft. in breadth), the difference in height between crest and trough is only 100 ft. Beneath this there is an older feeble but wide faulting to which the Seminole uplift belongs, the size of which is reflected in the extension of the oilfield.

The lower structure cannot be recognized from the surface geology. The sediments south of the Arbuckle Mountains are thicker, they apparently spring from them. After the deposition of the "Hunton" formation (Silurian) occurred the Seminole uplift, probably in the Devonian period. Subsequent erosion removed partially the "Hunton" limestone. After the deposition of the lower carboniferous the Seminole uplift was affected by slight faulting; erosion then occurred and members of the upper carboniferous series were laid down upon the consequent relief. To the west and north these members were thinner, but to the east and south thicker, conglomerate occurring also. After the upper carboniferous age the field was slightly lowered.

The "Hunton" limestone is only left on the edge of the uplift and in the south-west. Its oil is connected with a buried mound and lies in the upper

porous portions, into which it seems to have filtered from the lower carboniferous shales which lie above.

It is considered that the "Wilcox" sands in the lower Silurian, this most fruitful source of oil in the field, derives its oil from the green shales lying beneath them.

The concentration of oil is in the wide flat faults in the Devonian thrown into anticlinal and synclinal fold. This concentration succeeded the formation of the uplift previous to that of the slight faulting. The Wilcox sand huge production is characteristic of the Seminole oilfield.

PRODUCTION OF THE SEMINOLE OILFIELD IN ITS FIRST YEAR.

	Barrels.	Wells.	Average.	Daily production in barrels on July 22, 1927.
Seminole	43,305,028 ..	305 ..	3050 ..	90,160
Bowlegs	14,650,688 ..	163 ..	1630 ..	201,909
Earlsboro ..	8,637,760 ..	101 ..	1010 ..	170,873
Searight	8,220,759 ..	42 ..	420 ..	32,345
Total ..	74,874,235 ..	611 ..	6110 ..	495,287

J. E. M. H.

242. EARTH TEMPERATURES IN THE OILFIELDS OF HANOVER. By Dr. K. Herrmann, Celle. *Petr. Zeit.*, Vol. 24, No. 6. p. 241.

The distribution of temperature in the salt-domes of the oilfields of Hanover confirmed by a lowered geothermic temperature gradient, is very significant. The earth temperatures of the oilfields of Oberg and Nienhagen are cited as examples. The quantity of heat concerned is calculated from the geothermic temperature gradient and the co-efficient of expansion of heat of the rock. The quantity of heat passing through each square metre of the earth's surface in the course of a year was found to be about 200–300 kgm. cals. Since the passage of heat through the earth's crust is proportional to the geothermic temperature gradient this amount of heat increases considerably in geological space of time.

The question arises whether the heat proceeds from the salt-domes or from the oil layers. However, there are salt-domes with normal temperatures (Beienrode, Sperenberg) therefore the heat must separate as hot-springs or by the radio-activity of potassium and potassium salts according to Prof. Kinne. Also the development of heat by oxidation processes is not possible in deeply enclosed oil-bearing strata. A chemical change is suggested—*e.g.*, polymerisation, which is exothermic, then the density of the oil is simultaneously increased. The decreased density of light oil of Oberg contradicts this point. The petroleum in the oil-bearing strata is not chemically at rest, thus geologically the stoppage of definite directions of development of these changes is expected. This point is exemplified for several oilfields, especially in Hanover, where the deeper oil-horizons of lighter oil lead to the higher. The accumulated oil-bearing strata are primary and independent of one another; thus the lighter oils are geologically the oldest at the time. Höfer, with regard to other oilfields, maintains that the lighter oils arise metamorphically from heavier oils and the proportion of CH_4 homologues/naphthenes simultaneously increases. Hence free carbon is formed during methanisation. This has been confirmed in layers at Oberg.

It is also doubtful whether the heat emitted during long geological periods of time arises exclusively from the energy content of the hydrocarbons. This may be brought about by polymerisation or depolymerisation. The latter—

i.e., decomposition of high molecular hydrocarbons is endothermic, and the heat may be taken from the earth. This heat may be set free again later by polymerisation. Actually during submersion into greater earth depths the petroleum absorbs heat and forms molecules with a higher energy content, which is again set free as heat during and after tectonic ascent with reformation of molecules. However, the extent of this heat development in both directions caused by chemical change in definite petroleum layers and the extent of each in the observed heat development still remains undecided in the present condition of our knowledge of the chemistry of definite oil-bearing strata. W. S. C.

243. Geology of Amory, Mississippi, Gas Field. Dr. W. R. Jillson. *Oil and Gas J.*, 26–36, p. 58.

On October 5th, 1926, at depth of 2470 feet a large gasser, "No. 1 Carter," was brought in. This well produced when flush 4,552,860 feet open flow daily. It is about 6 miles east of Amory in Monroe County. On November 13th, 1927, a second well, "No. 1 Rye," was brought in. This is about 15 miles south-east of Amory. The well is reported to be producing from 3 to 4 million cub. feet of natural gas daily. With these two large gassers producing, and upwards of 10 fairly deep drillings in progress, regional exploration here is well under way. Geographically the field is located close to, if not exactly upon, the crest of the Cincinnati arch, which in this part is rapidly plunging along an axial line to the South 45° West. Surface rocks are Tascaloosa in age, sands, gravels and clays. Occasional outliers occur in the red Eutow sands. These unconsolidated sediments give little if any dependable criteria at the surface as to what detailed sub-surface structural conditions may be. Cretaceous and older Mesozoic sediments if present in this locality may range from 600 ft. to 700 ft. in thickness. There is apparently an angular erosional unconformity which is probably also a disconformity between the lower Mesozoic and the uppermost Palæozoic sediments. The record of No. 1 Carter gas well shows sands variously coloured between depths 985 ft. and 1468 ft., at 1495 ft. dark calcareous sandstone; then 25 ft. of clay; then shales and sands between 1590 ft. and 2160 ft., with some tar springs between 1555 ft. and 1865 ft. From 2200 ft. to 2470 ft. (the bottom) shales and limestones (mainly shales). At 2470 ft. the grey Hardinsburg sandstone is termed the "jett oil and gas sand." The oil production is considered by the author to belong to the upper part of the lower carboniferous. This "Hardinsburg sandstone" in Kentucky has been co-related with the "Hartsell sand" so petroliferous on outcrop in N.W. Alabama, and is the same as the chief producing sand of Northern Ohio Co.—viz., the "Jett," which produces daily about 6,500 barrels of petroleum. Several drillings are prospecting the Amory gas sand. The gas wells referred to at the beginning of this paper show that the "Hartsell sandstone" is now to be regarded definitely as an important producing horizon in north-eastern Mississippi. At points somewhat removed to the north-west from the axis of the Cincinnati arch immense quantities of petroleum may also be found in this important silicious bed. By further prospecting, other sandstones may finally be revealed to be quite as important oil and gas producers here on the eastern shoulder of the old gulf embayment as much farther to the north in western Kentucky. J. E. M. H.

244. The Geological Connection Between the Emba-Ural Oilfield and that of the Caucasus. *Pet. Zeit.*, 1928. 24,4 A. F. von Stahl.

On the ground of palaeontological research, Russian geologists consider that the oil-beds in the Emba-Ural field belong to the Jurassic period, which is esti-

mated at from 320 to 360 metres thick at Dos-Sor, but a little further east only reaches 100 metres. The petrographic nature of the Jurassic sediments however is against this. An attempt is made to sketch the tectonic occurrences by which this neighbourhood as well as the Caucasus were affected, thus obtaining the key to the possibilities of oil retention and their relationship between the oil occurrences of the Emba-Ural field and those of the Caucasus. The presence of Granitic and more ancient Palæozoic rocks, and other facts, indicate that in the Palæozoic age, a mountainous or elevated range stretched over the region of the Dneister, Bug, Ehaterinoslav and south of the Donetz coal basin in a south-easterly direction to the peninsula Mangischlak and probably also further to the hills of Serawschan, separating the Aral Caspian basin into a north-eastern and a south-western part. In the Carboniferous age the deposition of coal took place in the Donetz basin, indicating periodic uplift and depression of this part of the north-eastern soil, by which land and marine sediments were laid down until finally this area was raised above sea-level. In the south-west basin no coal deposition took place in the Carboniferous period and the sea appears to have been shallow.

In the Permian age an extensive shallow sea stretched from the Mangischlak hills northwards, being bounded on the east by the Ural range, and separated from the ocean, lost its water by evaporation, and was converted into a desert with numerous saline lakes and a strong saliferous earth. In gorges, deep basins and cavities accumulated salt which crystallised out and formed rock-salt. During this period petroleum may have been formed in places, such as are indicated at Kasan, Samara, and elsewhere on the Volga. The Triassic sea invaded this territory and covered large areas, but does not seem to have lasted long since its sediments are only feebly represented. It seems probable that the raised portion which had separated the north-eastern from the south-western area sank during this epoch, whilst the Caucasus Mountains rose above sea-level at the centre. In Jurassic times a sea extended over a wide area which in the central part of the Caucasus and in northern Persia were very deep, but shallowed towards the north-east. To the south-west and south coasts depression ensued during the deposition of the Lias and Dogger sediments, and mineral coal was formed. During the Cretaceous period an extensive deep ocean prevailed, both round the Caucasus and in northern Persia and the Trans-Caspian, to the Emba-Ural area and further north, forming massive sediments. In the Jurassic and Cretaceous ages petroleum may have been formed. In the Cacausus and Trans-Caspian or Persian area oil occurs in these formations, and it is very questionable whether in Jurassic times favourable conditions for oil formation were present in the Emba-Ural district. It is more probable that oil migrated from other formations into the Jurassic sediments, owing to powerful orogenetic movements, causing fractures and even tilting such as are found in the Emba region. An uplift and faulting of the Caucasus Mountains took place after the deposition of the Upper Cretaceous. The extension of Nummulitic Eocene to the south of this range indicates this tectonic movement.

Next the northern foot of the Caucasus sinks and the Oligocene waters flow in, washing the whole northern flank of the mountain from the Carpathian to the peninsula of Apscheron, Trans-Caspia, and the Emba-Ural area. Clay shales and marl-stones constitute the bulk of these sediments easily identified and co-related with those in the Caucasus. Such deposits as overlie these are of "Sarmatian" age.

Sandstones, sand and clay formation of the second stage of the "Mediterranean" series occur in the Caucasus in part clearly transgressing the Oligocene. "Pontian" beds are also seen. On Apscheron lie oil-bearing beds partly derived from the mainland, the oil being concentrated in the Sperialis

shales and Oligocene. Sarmatian and Pontian stages are not represented here.

By uplift of the Emb-Ural area the Caspian Sea was separated from the Sea of Aral and acquired its existing form. From these remarks it follows that only the Permian Sediments, perhaps Permo-Triassic, and Oligocene and Sarmation deposits can be oil-bearing rocks, though erosion has removed a great part of the last named and the oil has flowed from its primary receptacles, collecting partly in cavities and basins and oxidised into asphaltic forms. The oil in Permian sediments or in a few Tertiary depressions remains, and also in island-like Tertiary sediment still left. In the Trans-Caspian territory the oil-bearing rocks are of the same age as those south of the Caucasus, and also those on the Persian shores. Further research alone can trace the oil beds near Dos-Sor and Makat of the Emba-Ural district.

J. E. M. H.

Coal and Shale.

245. Developments in the Braunkohle Industry of Germany.—*Chem. Met. Eng.* 1927, **34**, 729–731. C. W. Cuno.

The total lignite production in 1926 was 150 million tons, most of which was obtained in open-cut working. The brown coal beds vary from 18 to 100 metres in thickness, the overburden being for the most part from 15 to 20 metres in depth. The raw material contains from 50 to 60 per cent. of moisture, its calorific value being 2,600 cal. per kg. The brown coal is utilised in three ways:—(*a*) as fuel without treatment, notably at several large electrical power stations erected by the A.E.G., sliding grates of a special type being used; or, after drying and crushing, in the pulverised form:—(*b*) as briquettes, this method of utilisation furnishing, approximately, 90 per cent. of the household fuel burned throughout Germany. In manufacturing briquettes, the coal is screened and crushed and then dried in rotary tubular steam driers, the moisture content being reduced to 15 per cent. The dried material is then cooled to 30° or 35° C. and briquetted without a binder at a pressure of 1,600 atm. Each machine is able to deliver 80 briquettes per die per minute, or, approximately, 100 lbs. The briquettes are smooth and compact, contain 5 to 6 per cent. of ash and possess a calorific value of 4,800 cals. per kg. (*c*) The brown coal is subjected to low-temperature carbonisation, the most recent of such methods being that of Kohlenveredlung A.-G. of Berlin. In this process the finely-ground coal passes between two vertical cylinders about 2 to 3 inches apart, each cylinder being alternately convex and concave in form, the inner cylinder being caused to rotate whilst the outer cylinder remains stationary. The latter is also lagged externally and is fitted with vents in each convolution for the withdrawal of volatile products. The furnace is a central combustion chamber much like a Bunsen burner, but having a down draft which carries the flames over a radiator of refractory material and then downwardly along the walls of the inner and rotating cylinder. The coal enters at the top, and the semi-coke is withdrawn at the bottom, the time of distillation being 18 minutes. A commercial plant deals with 100 tons of brown coal per day. The evolved vapours enter a tubular water cooler in which most of the tar is condensed, pass thence to a Cotterell, electrostatic separator to remove the remainder of the tar, after which the water is condensed and the benzol absorbed in light paraffin oil. The residual gas contains about 25 per cent. of carbon dioxide as well as hydrogen sulphide. It is bubbled through water at a pressure of 15 to 20 atm. to eliminate these

impurities. A half of the gas made is sold for domestic use. The tar is worked up for benzol, gas oil, fuel oil and paraffin: it will also yield lubricating oils. Balance and flow sheets indicate that low-temperature carbonisation of brown coal by this process may yield a considerable profit.

H. G. S.

246. The Cracking of Low-Temperature Coal-Tars. *Ind. Chemist*, 1928, IV., 109–114. A. E. Dunstan and H. G. Shatwell.

Two low-temperature tars obtained by the carbonisation in externally-heated retorts of (*a*) a South Yorkshire feebly-coking coal, and (*b*) a blend of a steam and a strongly-coking coal were subjected to examination with regard to their probable value as fuels and as materials for cracking.

Tar A1 (laboratory prepared sample), Tar A2 (large scale) and Tar B had the following respective properties:—Specific Gravity, 1·038, 1·035, 1·042: Tar-acid content 21 per cent., not noted, 26 per cent.: Volatile to 200° C. 11·5 per cent., 20·2 per cent., 12·5 per cent. The straight-run spirits were recovered, their contained tar-acids removed and the resulting hydrocarbons refined with acid and alkali, or acid and sodium hypochlorite or acid and a refining earth. The refining losses varied from 16 per cent. to 37 per cent., due largely to polymerisation of certain constituents, but the refined spirits were of good colour and odour and possessed very marked anti-pinking properties. Two pitch samples, respectively 44·35 per cent. and 34·35 per cent. residues of tar B, were also prepared, and were found to be of no value either for briquetting or road surfacing purposes.

Cracking experiments on various residues and distillates of the two tars were conducted in autoclaves specially constructed to allow of accurate temperature control, the object being to determine the conditions necessary to yield the maximum of spirit. Thus the residue boiling over 200° C. of tar A was heated to 441° C. for 70 minutes at a pressure of 550 lbs. per sq. inch, the yield of spirit to 200° C. being only 7·5 per cent. of the stock. The spirit contained 30 per cent. of tar acids. Cracking in two stages, the cracked spirit being distilled off after the first treatment, gave 12·8 per cent. of spirit which contained 33 per cent. of tar-acids. Distillation under 160 lbs. per sq. inch pressure at a maximum temperature of 451° C. yielded 12·6 per cent. of spirit containing 40 per cent. of tar-acids. The average yields of cracked spirit hydrocarbons for the three series of experiments were 5, 8·5 and 7·6 per cent. respectively. The 11·7 per cent. to 77·7 per cent. distillate of tar A (Sp. G. 1·040, I.B.P., 198° C., 48 per cent. over to 300° C.) was distilled at 480 lbs. per sq. inch pressure, the temperature rising to a maximum of 450°C. The results were:—Cracked spirit 14·8 per cent. (tar-acid content 30 per cent.): gas 80 volumes per volume of charge: coke 60 per cent. by weight.

Tar B was next dealt with. Two distillates (*a*) 4·45 per cent. to 51·2 per cent. (*b*) 5·65 per cent. to 60 per cent., *i.e.*, the crude tar with spirit to 170° C. and pitch removed, were combined and a portion extracted with caustic soda to eliminate tar-acids. The latter were subsequently recovered and three cracking stocks thus prepared, *i.e.*, 1 neutral oil and phenols, 2 neutral oils, 3 phenols. The three materials had the following respective properties: Specific gravity 0·992, 0·956, 1·055: tar-acid contents 34 per cent., nil, 100 per cent. Each stock was subjected to distillation at pressures varying from 240 to 325 lbs. per square inch. The yields of products in comparable experiments in which distillation occupied 140 minutes for each stock were respectively: crude spirit 31, 26·1, 36·2 per cent.: tar-acids in spirit 34, 4, 100 per cent.: gas volumes per volume of stock 69·3, 63·8, 69·6: coke by weight 21·2, 20·2, 23·6 per cent. The quantity of cracked hydrocarbon spirit

produced approximated to 20 per cent. of the stock or 11·12 per cent. of the crude dry tar.

The above experiments utilised charges of 300 c.c. of cracking stock but a larger autoclave capable of dealing with one gallon of material was used for the following. Two batches of tar A were first distilled to 200° C. to eliminate spirit and the distillations continued to leave 34 per cent. and 27·4 per cent. residues respectively. Tar B, was similarly dealt with giving an 8·2 per cent. to 57·8 per cent. distillate from which tar-acids were extracted prior to cracking. The three materials were distilled under pressures of 150 to 185 lbs. per square inch, the time from first to last drop being 4·25 hours in each case. Persian fuel oil was distilled under similar conditions to obtain comparable figures. The results are tabulated in detail, the following being the most important data taken in order of 1 tar A, 20 per cent. to 66 per cent. distillate; 2 Tar A, 20 per cent. to 72·7 per cent distillate; 3 Tar B, heavy hydrocarbons in the 8·2 per cent. to 57·8 per cent. distillate; 4 Persian fuel oil. Pressure distillate per cent. by volume of stock 64·3, 48·2, 67·5, 73·6; crude hydrocarbon spirit 16, 11·8, 24·2, 56·7 per cent.; tar-acids 16 per cent., 19·4 per cent., nil; gas volumes 71, 112, 93·2, 94; coke by weight 29·2, 42·0, 24·0, 20·9 per cent.: spirit hydrocarbons on crude tar or oil, 7·7, 6·2, 6·56, 36·8 per cent. The cracked spirit from tar B refined with 1 per cent. of strong sulphuric acid and alkali was stable to light and possessed an anti-knock efficiency of 40 (benzol = 100). The cracked spirit from tar A when refined with 1 per cent. of acid was not stable but with sodium hypochlorite and 0·5 per cent. of acid, a pale yellow but sweet-smelling spirit was obtained. Its anti-knock benzol equivalent was 50. It is concluded that low-temperature tar residues and distillates are very inferior cracking stocks, and that the possibility of utilising them for such purposes on a commercial scale, bearing in mind the difficulty of separating and disposing of the tar-acids, would be problematic. On the other hand, the cracked spirits can be refined by conventional methods and they possess valuable anti-knock properties.

H. G. S.

247. Distribution of Sulphur in Oil Shale—II. Harding and Dumke. *Ind. & Eng. Chem.*, 20, II. 164.

The paper is a continuation of another of the same title.

Three shales have been examined and the following constants determined :—

	El. Rs. Nevada.	Green River Utah.	Green River, Mt. Logan.
	%	%	%
Total sulphur	4·946	1·101	1·373
Sulphide and sulphate sulphur	3·998	0·556	0·724
Sulphate sulphur	0·406	0·146	0·6375
Sulphide sulphur	3·592	0·410	0·0865
Organic sulphur	0·948	0·545	0·649
Humus sulphur	0·948	0·540	0·644
Resinic sulphur	0·000	0·005	0·005

A. R. S.

248. Motor Fuels from Coal. A. E. Millre, New York. *Petr. Zeit.*, Vol. 24, No. 3, 103.

The author considers the following methods to be promising in the production of motor fuels from coal or lignite :—

1. The distillation of pulverised bituminous coal or lignite by blending heavy oil residues or tar under great pressure at high temperatures and hydration with or without catalysts.

2. Low or high temperature distillation of coals producing all the valuable liquid and gaseous derivatives down to coke and the consequent production of water-gas from coke. The water-gas, after removal of sulphur compounds provides the crude material for two types of a catalytic process :—

(*a*) Hydration under high pressure and at high temperatures in the presence of certain catalysts in the production of synthetic alcohols.
(*b*) Hydration at atmospheric pressure at a moderate temperature in the presence of definite mixtures of catalysts in the production of synthetic hydrocarbons.

Examples of these methods are cited :—

1. *Bergius Process* and a process elaborated by the Bad. Anilin and Soda Fabrik usually designated as the B.A.S.F. process.

2. *Patart process*, methanol process (Franz Fischer) and the B.A.S.F. process for the production of synthetic alcohols.

3. *Franz Fischer's process* for the production of synthetic hydrocarbons.

The author gives in the following a short description of the above-mentioned processes.

The Bergius Process.

Dr. F. Bergius utilises heavy petroleum—or coal-tar residues, coals rich in bitumen, or lignite, Fe_2O_3 and H_2 as initial materials. The finely-powdered coal is mixed with the oil and Fe_2O_3 to a thick paste and placed in a double-walled retort under hydraulic pressure and kept in motion by a stirrer. The temperature is maintained at about 800–900° F. by passing a stream of gas at this temperature through an iron tube plunged in molten lead and thence circulated through the double wall of the reaction chamber. The whole heating system is closed. The gases are utilised, after they have been applied in the heating of the distillation gases, to preheat the H_2 which is introduced into the retorts whereby they cool to a temperature at which they are again compressed, and can be led back after repeated heating into the heating space of the retorts. Thus the hot gases are never in direct contact with the gaseous or liquid products in the reaction chamber. The pressure maintained in the reaction chamber amounts to about 150–200 atms., and is a little greater than this in the heating space. The volatile products and gases escape through an outlet and are then fractionally distilled and treated in a gas-separator. The slag-like mixture, the coal-ash and the Fe_2O_3 are removed from the retort by means of a screw-stirrer and collected in suitable containers.

The coal is not completely liquefied in a single process through the Bergius retort but three such throughputs are often necessary to give a complete yield. According to Bergius two different reactions ensue. Firstly, addition of H_2 principally to the coal itself, secondly cracking whilst fresh quantities of H_2 are admitted.

Dr. Bergius maintains that catalysts play no part in his process, but that it only involves a reaction occurring at high temperatures and under high pressures between heavy oils or oil-coal mixtures, with simultaneous cracking and hydrogenation. Miller is opposed to this view. However, Bergius states that the Fe_2O_3 present in the paste of coal and oil acts to a certain degree as a catalyst. Bergius' products consist of aliphatic, aromatic and hydro-aromatic hydrocarbons, also phenol, cresol, NH_3 and organic N_2 compounds, but not of alcohols.

About one ton of oily crude products is obtained from three tons of coal in the Bergius process—thus, the coal consumption for power and heating is very high. Also the benzine fraction is greatly diminished after removing phenols and N_2 compounds.

M. Heyn and M. Dunkel have shown that in the removal of phenolic bodies from benzine or from higher fractions up to 220° C. (428° F.), a loss of > 16 per cent. is produced.

The B.A.S.F. Process for the Production of Synthetic Alcohols.

This is a modified hydrogenation process performed at high temperature and pressure and in the presence of catalysts. A mixture of H_2 and CO in the presence of excess CO is brought into reaction under a pressure of about 120 atms. and at a temperature of 400° C. (752° F.) in the presence of certain catalysts, *viz.*, asbestos, pumice-stone or other porous substances which are impregnated with cobalt oxide or zinc oxide and a small quantity of NaOH or Na_2CO_3. The products obtained consist of an oily layer comprising two-thirds saturated and one-third unsaturated, hydrocarbons, also an aqueous layer containing a mixture of various substances which do not resemble homogeneous saturated hydrocarbons rich in petroleum.

F. Fischer's Process.

F. Fischer and Tropsch modified the B.A.S.F. by utilising an excess of H_2 instead of CO. The reaction products consisted of an oily layer containing the majority of the higher alcohols, ketones and aldehydes (synthol), and an aqueous layer. These products resembled those obtained by Patart.

Fischer's methanol process consists of a hydration of CO under high pressure in the presence of zinc oxide as a catalyst. Water-gas or other gaseous mixtures which contain CO and H_2 are first of all freed from sulphur compounds and introduced into the reaction tube. This is 30 in. long and 3 in. in diameter and is made of glass, aluminium or copper, and heated to 482° F. The catalytic mixture in the reaction tube usually contains finely divided metals like Fe and Co, or Fe and Cu, also bases of different strengths like Na_2CO_3, $Ba(OH)_2$, Li_2CO_3, etc. The reaction can occur at very low pressures, at atmospheric pressure or less than atmospheric pressure. The reaction tube is connected with several condensers, thence connected to an absorption tower filled with charcoal.

Fischer obtained three fractions from his gas mixture and oil-vapour mixture in the reaction chamber, namely, a heavy fraction consisting of paraffins and oils resembling lubricating oils; a light oil fraction and a light benzine fraction. The heavy fraction was bright yellow, the kerosine fraction was not absolutely "water-white" but the benzine was quite "water-white." The two heavier fractions were obtained directly by condensation whilst the gasoline was collected in the absorption-tower and then expelled with steam. The reaction appears to proceed thus: One molecule CO_2 arises from two molecules CO under the existing conditions. The second hydrocarbon atom unites with the metal catalyst forming a carbide which is unstable.

By careful choice of the strengths of alkaline bases which are mixed with the metal catalyst the degree of polymerisation can be regulated exactly so that all desired hydrocarbons from benzine to paraffin can be produced.

Since the crude substances reacting according to Fischer, contain no sulphur, the products are of very special purity. Thus tar or coke does not

arise, only pure hydrocarbons besides the non-reacting gases. This circumstance gives Fischer a significant advantage over Bergius.

W. S. C.

249. MOTOR FUELS FROM COAL.—By A. E. Miller, New York. (*Concluded.*) *Petr. Zeit.*, Vol. 24, No. 4, p. 139.

G. Patart's Process.

This method is based on a direct synthesis from CO and H_2 under pressure and in the presence of a catalyst. The gaseous products obtained from the complete gasification of coal are utilised as the crude material. After the production of water-gas the majority of the sulphur-containing compounds are removed by a special method. A pressure of 300 atmospheres is applied to the gases, which then pass into the catalytic chamber where they are subjected to the action of certain catalysts at the above-mentioned pressure. If methanol is desired, temperatures between 662° F. and 752° F. are applied, and if higher alcohols are desired, a temperature between 752° F. and 840° F. is maintained.

Patart discovered that the best mixtures of zinc oxide and chromium oxide in appropriate proportions were suitable for the synthesis of alcohols. These mixtures of catalysts are found to be less sensitive to the influence of "catalytic poisoning," and the regeneration of the catalysts is quite simple. The application of such high pressures is necessary because the total pressure for the synthesis of alcohols does not come into consideration but only the partial pressure of the gases reacting—*i.e.*, of CO and H_2. The greater the percentage of the enclosed gases the greater must be the total pressure. The reaction chamber in which the catalyst is present is constructed of copper, aluminium, chromium or of any substance unattacked by H_2 and CO_2—iron must be avoided. According to Miller the Patart process proceeds thus :—The purified gas which generally contains 80 per cent. of gases capable of reacting enters the catalytic chamber under the temperature and pressure conditions mentioned, and is then subjected to fractional condensation. The various products are then collected in receptacles, whilst the non-reacting gases are again led back into contact with the catalyst. Thus the content of non-reacting gases increases since CH_4 is formed and the N_2 content is enriched. The circulating gases have to be freed from these non-reacting gases, otherwise the yield is small and the pressure has to be greatly increased. The products obtained by this method are alcohols, acids free or bound, ether, esters, aldehydes, ketones, and hydrocarbons of all kinds. The only compound which could be obtained in the pure state was MeOH, which could be produced in almost theoretical yield. One ton lignite can yield at least 880 lb. methyl alcohol. Patart states that 1 lb. methyl alcohol should be obtained from every 2 lb. coal rich in lignite. In the case of higher alcohols the yield is less—*e.g.*, only 1 lb. of isobutyl alcohol can be obtained from 3 lb. of coal. Hydrocarbons can be obtained from the higher synthetic alcohols by reduction, using aluminium as catalyst. Patart considers that by his method hydrocarbons can be produced economically, as in the direct hydration of coal. The apparatus, producing 20 tons MeOH/day would cost between 400,000–500,000 dollars. The greatest outlay is for compressors to produce at least 500 atmospheres pressure. For the production of 1 lb. methyl alcohol about 1–1·27 kilowatt-hrs. energy is required, and ·1·68–2·2 kilowatt-hrs. energy for the production of 1 lb. isobutyl alcohol. A crude substance containing 50 per cent. water-gas is reasonable. Patart assumes that MeOH produced by his process would cost about 20 cents/gallon in the

pure state. Then MeOH would be about half the value of benzine corresponding to about 40 cents gal. motor fuel.

The Present Condition of the Processes Described: Bergius Process.

Miller states that a large plant is under construction in the Ruhr territory which should produce 60,000 tons of synthetic oil per annum, and with favourable results the same should be increased to 400,000 tons—*i.e.*, about 2,500,000 barrels of oil.

F. Fischer's Process.

The erection of a research institute near Essen for the development of Franz Fischer's Process is anticipated. This medium would utilise the Ruhr coals and the Rhine lignite syndicate.

It appears that in the near future three methods for the production of motor fuels from coal will be tested on a large scale in Germany :—

1. B.A.S.F. Process for the production of Methanol.

2. High-pressure hydration process developed by the B.A.S.F. for the production of mixtures of hydrocarbons and alcohols—similar in certain points to the Bergius process.

3. Franz Fischer's Process for the production of synthetic hydrocarbons.

Miller states that Fischer's process has claimed great attention, and further important results of research in this direction are anticipated.

250. Contributions to the Science of Natural Bitumen with special Consideration of Bituminous Shale-Peat. By Dipl. Eng. W. Zwieg, Karlsruhe. *Petr. Zeit.* Vol. 24, No. 6, p. 243.

The author describes a type of peat found in various moors of N. Germany which is present at the base of the ordinary high moor peat, and resembles cannel coal. It is characterised by a significant content of bitumen and turns black on drying in air. On account of the shale-like structure of the air-dried substance it is designated in Mecklenburg as "shale-peat," *i.e.*, tar-formers, like wax, resins and fats.

Potonie differentiates between (1) *Shiny or Humus coals* and (2) *Sapropel or dull coals*. To (1) belong pit-coals, lignites and ordinary peats, and to (2) belong bituminous fuels, *e.g.*, oil-shales, cannel coals and other similar coals. The geologically youngest members of the Sapropelite group is found in the slime from stagnant waters and consists of a pulp-like structureless mass which changes in time into a dark-brown jelly-like substance, *Saprocoll* ("bad jelly"). The latter is very thick and hard in the air-dried condition, and has the property of opening like shale when dried quickly in the sun.

The original matter of the Sapropel coals passes through a process of putrefaction called "bitumination" during which the proportion of hydrogen to carbon remains the same.

The percentage of carbon and hydrogen in the ash- and water-free condition of Humus and Sapropel coals is given in tabular form. These tables show that the proportion of C/H in all oil-shale minerals is constant and remains constant with increasing geologic age. Thus the shale peat in question obtained from a W. Prussian moor appears to be different from the ordinary high moor peat. Its synthesis is briefly described. The layer of a diluvial trough of the moor consists of bluish-green clay of about 1.5 m. depth covered by a strong layer of about 1 m. of alluvial mud which "shuts off" the moor from the ground-water. Above the clay mud lies about 3 m. of strong shale-peat which differs from the overlying high moor

peat layer by its homogeneous property. When freshly obtained the material has a water content of about 90 per cent.

The analysis of finely powdered air-dried shale-peat gave the following results :—

	Composition of :— Crude coal.	Pure coal.
Ash	4·81%	
Water	13·94%	
Pure carbon	81·25%	100%
Coke yield	18·84%	
Pure coke	14·03%	17·25%
Volatile constituents	67·22%	82·75%
Nitrogen	2·89%	3·56%
Total sulphur	0·32%	
Combustible sulphur	0·27%	0·33%
Maximum calorific value	4·420 k./cal.	5·440 k./cal.
Minimum calorific value	4·047 k./cal.	
Bitumen	20·4%	25·1%

On account of the easy oxidisability of the dry substance, the water content was not determined by drying at 105°, but by distillation with tetrachlorethane (Tausy and Rumm's method). Zwieg states that the water contents found by the usual method for water determination for oil-shales amount to about 1 per cent. under the actual value. The adoption of a method of determining humidity by distillation with xylol is recommended in preference. The nitrogen content follows from the usual Kjehlahl method.

The extraordinarily high N_2 content of the pure coal appears to be a characteristic sign of bituminous shales. The coking of shale-peat confirms a considerable bitumen content but not oil-bitumen. The solid bitumen has a very low decomposition point. The coke yield itself is low and the content of volatile constituents is high. The latter is constant but with increasing geologic age and progressive bitumination of the Sapropelite the content of disposable hydrogen naturally increases.

Air-dried shale-peat gives an essentially high calorific value. An elementary analysis gives :—C 44.7 per cent., H 5·3 per cent., O_2 28·0 per cent., N_2 2·9 per cent., S 0·3 per cent., ash 4·8 per cent., and H_2O 13.9 per cent. W. S. C.

251. A Process for Recovering Liquid Products from Coal. H. Novak and A. Brod. E.P. 283,269, 1926.

Coal, brown coal and the like is distilled under pressure in an autoclave in presence of an aromatic hydrocarbon the boiling-point of which exceeds 230° C. The temperature of the coal-oil mixture is raised gradually from 200° to 500° C. and the distillation so conducted that the low-boiling products distil off first, followed by the aromatic hydrocarbon. The heavy oils remaining in the autoclave are next cracked at a temperature exceeding 380° C., the final stage being the dry distillation of the solid residue at 500° C. The aromatic hydrocarbon consists of liquid homologues of naphthalene or mixtures thereof with solid hydrocarbons or of fractions of coal-tar or generator tar. In the example, 1 kg. of Bohemian brown coal (7·8 per cent. moisture, 6·8 per cent. ash) and 2 kg. of anthracene oil (250° C. to 300° C.) were distilled at 25 atm. pressure in an autoclave fitted with a reflux, the latter being cooled in its upper portion. The anthracene oil was recovered un-

changed, the yields of products calculated to coal used being:—Benzine (60°–180° C.) 8 per cent., oils 10·0 per cent., aliphatic tar 26·8 per cent., semi-coke 43·4 per cent., water 7·8 per cent., gas and loss 4·0 per cent.; total 100·0 per cent. H. G. S.

252. Process for the Distillation of Solid Fuels at Low Temperatures. Compagnie des Mines de Bruay. E.P. 272,522, 1928.

The fuel to be distilled is briquetted and then heated within a mass of ponderous material, each briquette being separately embedded in the mass which may consist of grains of inert materials such as silica, magnesia, kieselguhr, common sand, metal filings and the like. Prior to briquetting, the whole or a portion of the powdered fuel may be dried and then moistened with a water solution of an agglomerating substance. The briquettes are dropped upon an endless conveyor covered with a part of the mass of granulated material, the remainder of the latter being then poured on to the conveyor. The distillation utilises a range of temperatures predetermined by the nature of the fuel and is preferably effected in a tunnel oven. The range of temperatures for a soft coal are (*a*) 100° to 420° C. in 20 min., (*b*) 420° to 460° in 1 hr., (*c*) 460° to 560° C. in 45 min., (*d*) constant heating at 560° for 15 min., (*e*) cooling from 560° to 400° C. for 40 min. H. G .S.

253. Improvements in and relating to Processes for the Distillation of Solid Fuels at Low Temperatures. Compagnie des Mines de Bruay. E.P. 282,340, 1928. Addition to E.P. 272,522.

The agglomerated pieces of solid fuel are heated in a mass of ponderous substances which will produce gases and oils when subjected to the process. The materials specified are dust from coal schist, bar coal, mined coal, roof coal or waste peat or vegetable wastes, *e.g.*, sawdust or dried oil-cake; or the solid fuel may be pulverised and mixed with powdered charcoal before briquetting. H. G. S.

254. Method of and Apparatus for the Distillation of Oils from Oil Shale or Coal or similar Material. R. H. Crozier. E.P. 283,639, 1926.

An externally heated, vertical retort provided with inclined flues traversing the retort is used to treat coals or shales of an intumescent character. Heat is supplied to the top of the retort so that the material to be distilled is at once subjected to a high temperature, *e.g.*, 400° C., the pitch-like constituents being volatilised, and a semi-coke, which will readily travel downwardly, being simultaneously produced. The hot gases of combustion are made to pass through a succession of external and internal flues to the bottom of the retort and are then taken in an upward direction through flues surrounding the inner flues. Means are also provided to supply heat to intermediate portions of the retort. H. G. S.

255. Improved Process for the Extraction of Petroleum Products from Bituminous Material. P. Dvorkovitz. E.P. 285,564, 1928.

The bituminous material is heated to a predetermined temperature to distil off a portion of the organic matter and then a solvent is added in the form of a liquid or vapour to dissolve the organic matter still remaining in the raw material. The solvent may be petroleum or gas tar. As example,

a bituminous coal containing 30 per cent. of organic matter is heated to 500° C. Petroleum or gas tar to the amount of 20 to 40 per cent. is then added. H. G. S.

256. Improvements in the Manufacture and Production of Valuable Liquid and other Hydrocarbons and Derivatives thereof by the Destructive Hydrogenation of Mineral Coal and other Solid Carbonaceous Materials. I. G. Farbenind. A.-G. E.P. 256,964, 1926.

In the hydrogenation of coal and other solid carbonaceous substances, except brown coal and peat, at elevated pressures, coking of the material frequently occurs. This can be obviated by mixing with the coal, etc., a certain proportion of brown coal or peat or both, usually about 20 per cent. This mixture behaves like a highly viscous liquid when under high pressure so that suspension of the coal in oil prior to admission to the plant becomes unnecessary. Catalysts may be employed, either mixed with the initial material or arranged in a separate reaction vessel or both. H. G. S.

257. Improvements in the Production of Liquid Hydrocarbons and other Products by the Destructive Hydrogenation of Coal, Tars and like Carbonaceous Materials. I. G. Farb-ind. A.-G. E.P. 256,965, 1926.

The raw materials in a gaseous or finely divided liquid or solid state are treated with hydrogen or gases containing or giving rise to hydrogen at high temperatures and under pressures exceeding 50 atm. in presence or absence of catalysts. The hydrogen or other gases is used over again in a circular course, the hydrogen consumed being made up by the addition of a mixture of hydrogen and water vapour or of water vapour alone, the latter not being employed in so great quantities that its partial pressure exceeds that of the initial carbonaceous material while exposed to the working conditions. The initial material may be conveyed through several reaction vessels arranged in series or circulated while removing the residues and making up for the portion converted. H. G. S.

258. Improvements in the Manufacture of Valuable Liquid and other Hydrocarbons and Derivatives thereof by the Destructive Hydrogenation of Carbonaceous Materials. I. G. Farbenind, A.G. E.P. 257,256, 1926.

The process comprises the manufacture of valuable liquid products from carbonaceous materials by the action of gases containing hydrogen and oxides of carbon, or of water vapour, under a high pressure and in presence of a contact mass capable of forming methanol by the catalytic hydrogenation of oxides of carbon. The contact masses employed may be those specified in E.P.'s 229,714, 229,715, 237,030 and 254,760, or the following: vanadic acid, manganese or uranium oxides, titanic or chromic acids, zinc oxide alone or mixed with the oxides of uranium, lead, chromium or manganese or with titanic acid. The contact masses are preferably arranged in a separate contact vessel through which the initial materials are passed in a gaseous or finely divided state together with the hydrogen containing oxides of carbon. When working with, e.g., water-gas, benzine and other hydrocarbons are formed, in addition to methanol and higher oxygen-containing compounds, the products being suitable as "anti-knock" motor fuels.

H. G. S.

259. Improvements in the Purification of the Liquid Complex Hydrocarbon Products of the Destructive Hydrogenation of Carbonaceous Materials. I. G. Farbenind. A.G. E.P. 257,270, 1928.

The method of purification consists in treatment with lower alcohols of the aliphatic series, e.g., crude synthetic methanol, preferably by a counter-current method. Example I: the product obtained by destructively hydrogenating a brown coal producer tar is freed from benzine by distillation and wax by chilling and pressing and then treated with methanol. All tarry oxygen-containing components are dissolved, whilst a 40 per cent. yield of hydrocarbons is left as residue.

H. G. S.

260. Improvements in the Manufacture of Liquid and other Hydrocarbons and Derivatives thereof by the Destructive Hydrogenation of Coal, Tars and the Like. I. G. Farbenind. A.G. E.P. 257,910, 1926.

The formation of coke in the process of hydrogenation can be eliminated by employing the raw materials in a moist state or by adding water during the course of the hydrogenation. H. G. S.

261. Manufacture of Improved Catalysts. I. G. Farbenind. A.G. E.P. 286,123, 1928, Addition to E.P. 281,218.

The metal catalyst is separated from a solution of its salt by means of a more electro-positive metal, or by means of hydrogen, under pressure if desired, in presence of an organic base or acid amide. Thus (*a*) 280 parts of cobalt sulphate dissolved in 1,000 parts of water are mixed with 240 parts of urea. There are then added at 60 to 70°C. 110 to 120 parts of zinc dust, stirring being continued until the precipitation of cobalt is complete, the temperature being maintained at 90° to 95°C. The precipitate is filtered and washed, (*b*) a nickel catalyst is prepared by treating a mixture of nickel sulphate, water, pyridine, kieselguhr, and finely divided nickel with hydrogen at 30 to 40 atm. pressure at about 100°C. H. G. S.

262. Improvements in Apparatus for Distilling Carbonaceous Materials. C. A. Griffiths. E.P. 228,749, 1924.

Solid carbonaceous materials are heated in a rotary retort of small diameter and having thin walls, the heat being applied externally. The walls of the retort are cleaned and heat distributed evenly through the material by the use of metal brushes, masses of tangled wire, chains, rings, discs or a screw conveyor. H. G. S.

263. Improved Method and Apparatus for Low Temperature Distillation of Coal. Int. Combust. Engin. Corp. and W. Runge. E.P. 267,095, 1927.

The process consists essentially in distilling a stream of finely powdered coal in a vertical retort by means of ascending hot gases, a portion of the heat contained in the latter being derived from the hot semi-coke leaving the plant. The combustible gas produced in the process, after separation from tar, is used to propel the coke through a cyclone separator to a storage bin, the gas being thereby preheated and the coke substantially cooled. The preheated gas is next further heated in a preheater of special design. A portion of the gas is burned in a combustion chamber, the remainder being taken through passages surrounding the preheater. The burnt and the

unburnt gases are then commingled in a mixing chamber from which they enter the base of the retort and pass in counter-current to the descending coal. More than 50 per cent. of the heat of the coke is recovered and at least 20 per cent. less fuel is required to operate the system. H. G. S.

264. Treatment of Carbonaceous Materials. E.P. 282,874. September 17, 1926.

A process is described for the separation of carbonaceous materials in bulk, especially coal, into heavier and lighter fractions by causing the lighter fragments to float in a liquid of specific gravity greater than that of such lighter fragments but less than that of the heavier fragments. Calcium chloride solution with an alkaline reaction has been found a suitable medium. The material to be separated is continuously supplied to the bath, the float and sink continuously removed from one another, and the sink washed by upward displacement and the float by downward displacement with a continuous unbroken body of liquid. It is preferable to remove the greater part of the dust from coal prior to the flotation treatment. The invention is particularly applicable to the separation of coal into a clean coal portion having a low proportion of ash forming bodies and a mineral matter portion having a high proportion of ash forming bodies. The float comprising essentially clean coal is removed from the top of the vessel, and the sink comprising essentially the mineral matter is carried out by an elevator, each into their respective drainers.

A preferred form of apparatus consists of a separating vessel which contains a solution of calcium chloride of suitable specific gravity which, for the separation of bituminous coal into constituents of high and low ash content, may be 1·35. Coal is fed by means of a screw conveyor to a hopper situated above the vessel, and is delivered through a chute which terminates about midway between top and bottom of the vessel. At the lower end of the chute a paddle agitates the bath at the point of introduction of the coal. The particles floating upward are removed by a revolving blade on to a sloping launder and are delivered to a shaft or tower where washing by downward displacement takes place. The sink collects at the bottom of the separating vessel and is removed by a bucket elevator to a tower similar to the above, where it is washed by upward displacement. Suitable pipes are arranged to collect the concentrated calcium chloride solution and washing water with the minimum loss of the former. The process may be performed intermittently or continuously, in the latter case a number of towers being provided and are successively connected to the bath for charging. After one tower has been changed it is washed and drained during which periods other towers will be charging.

Other substances than calcium chloride may be employed, and in a modification of the process the washing may be incomplete so as to leave a predetermined quantity of solute in admixture with the coal, so that it will act as a catalyst during subsequent carbonisation, combustion, hydrogenation or other utilisation. H. S. G.

See also Abstracts Nos. 166, 171, 172, 173, 186, 188, 217, 223 and 230.

Abstracts.

Arranged by W. H. THOMAS, A.R.S.M., and H. G. SHATWELL, M.Sc.(Tech.), Ph.D.

Abstractors—S. F. BIRCH, Ph.D., D.I.C., Miss W. S. CLARKE, B.Sc., S. W. COOKE, B.Sc., P. DOCKSEY, B.A., H. S. GARLICK, B.Sc., A.I.C., J. E. MARSHALL-HALL, A.R.S.M., W. N. HOYTE, B.A., G. SELL, H. G. SHATWELL, M.Sc.(Tech.), Ph.D., E. R. B. SKIPWITH, B.A., T. A. SMITH, B.Sc., A. R. STARK, B.Sc., A.I.C., W. H. THOMAS, A.R.S.M., T. WISKIN

General.

265. THE PETROLEUM INDUSTRY IN FOREIGN COUNTRIES. *Petr. Times*, 1928, XIX. (483), 675-676.

In a paper ("The Economic Outlook for Exports of Petroleum Products") before the American Institute of Mining and Metallurgical Engineers, Mr. John H. Nelson deals with the tendency to control the petroleum industry by legislative enactments.

By a Royal Decree of October 20, 1927, Spain has granted an exclusive monopoly for importation, storage, distribution and sale of all oil products to a Banking Corporation. Oil properties in Spain of other nationals are to be expropriated and owners reimbursed.

In France an alternative to a monopoly has been suggested in the form of regulation of imports by licensing.

In Italy the Azienda Generale Italiana Petroli is a state-subsidised oil company, its objects being to "assist" private enterprise and to control operations of foreign companies by selling oil at reduced prices.

Russia represents complete nationalisation, about 30 per cent. of the national income being secured from oil.

In Rumania only Rumanian citizens can hold titles to oil properties, but the elemental fact that the oil industry requires large capital, which Rumania lacks, has been ignored.

The Cartel system is preferred in Poland, and a new centre was established on December 10, 1927.

Greece has had a kerosine monopoly for years, and Turkey has a petroleum monopoly which prohibits the functioning of the law of supply and demand.

Japan is discussing the possibilities of governmental control of production and sales, and the Chinese Nationalists are favourably disposed to a kerosine monopoly.

In Australia the refining and sale of oil products is subsidized through the Commonwealth Oil Refiners Co., Ltd., at considerable public expense.

The control of the oil industry is apparently being readjusted and Chile, by an amendment of the National Mining Code, reserves all petroleum deposits to the State, claims not developed before December 26, 1927, being cancelled. A recent Bill provides a duty on Diesel or Semi-Diesel of 3 pesos

per gross ton of 100 kilos until December 31, 1928, and of 3 pesos more each year up to 21 pesos per ton, this figure then to remain in force permanently.

In the Argentine a general scheme exists for the products from the Government refinery, and there is a tendency towards nationalisation of production.

Brazil now has a Bill in Congress which includes a number of Federal regulations covering petroleum exploration and development. G. S.

266. Modern Ideas in Fractional Distillation. C. S. Robinson. *J. Soc. Chem. Ind.*, 1928, **47**, 405-8.

After a brief consideration of the theories underlying the distillation of binary mixtures, the problem of the design of a fractionating still is dealt with. This lies in the design of the rectifying column and the supply of sufficient heating and condensing elements, all of which factors are closely inter-related. The design of the column consists in the determination of the number of plates of the most suitable type or when filling materials are used, the height and diameter most fitted to fulfil the conditions. Consideration must also be given to the concentration, temperature and rate of feed of the liquid to be distilled, the composition of distillate and residue, the temperatures and quantities of the cooling and heating mediums, the cost of heat, water, labour, etc. The method of calculating the number of plates required was devised by McCabe and Thiele (*Ind. Eng. Chem.*, 1925, **17**, 1925). Although the design of a fractionating still for complex mixtures is much more complicated, it is possible to generalise from the solutions of problems involving the distillation of binary mixtures. The following generalisations are drawn from the study of binary mixtures and are directly applicable to mixtures of any complexity. (1) The function of that part of the column of a continuous still above the feed plate is solely to strip the less volatile from the more volatile components of the distillate. (2) The function of the lower portion of the column is to remove the more volatile component from the less volatile residue leaving the bottom of the column. Therefore the feed (in a continuous still) should be introduced into the middle, and not the top or bottom, of the column. (3) Effective working of any part of a rectifying column is impossible without a supply of heat below and a removal of heat above the point in question. Heat absorption may be due to cold feed, low-boiling feed, or down-flowing feed or reflux, whilst the heat supply below may be due solely to the latent heat in the vapour coming from some entirely separate apparatus, or directly supplied from the heating surface at the base of the column. (4) In general, it is more economical to use a single condenser at the top of the column and to return part of the total condensate as reflux, using a somewhat higher column, than to try to obtain additional rectification by more complicated condenser design. (5) In general, the permissible vapour velocities in a column are substantially independent of the composition of the mixture being distilled, no matter how complex. (6) Except under very special conditions, it is impossible to make more than one sharp cut per column. A continuous still must have as many columns, less one, as there are products into which the original mixture is to be separated. (7) If a known mixture is to be separated sharply into two fractions at some specific point, the temperature and pressure at both the top and bottom of the column are thereby automatically fixed. (8) Additional cooling at the top will produce additional reflux, but if separation is good, and the temperature in the condenser is not changed, the composition of the product will remain unchanged. (9) When a partial condenser is used in the removal of a mixture of several components as the distillate, the outlet temperature of this condenser is always the condensing temperature of the distillate under the operating pressure and is always intermediate

between the initial and final boiling points of the distillate. (10) The initial boiling point of a mixture is always above the boiling point of the most volatile pure component, and the final boiling point is always below the boiling point of the least volatile pure component, unless that component is present in excess. (11) It is practicable to feed a column in a continuous still with either liquid or vapour or any mixture of the two. Feeding with liquid tends to overload the lower part of the column. Feeding with vapour overloads the column when the percentage of distillate in the feed is small, but may be very desirable when the percentage of distillate in the feed is large. (12) If the feed enters as liquid, and a small percentage is removed as distillate, the main task of the column is to strip all the volatile constituents from the residue, which means that a large exhausting section is required. Conversely, a large column above the feed pipe is required when most of the feed is taken off as distillate. H. G. S.

267. Royalties in Petroleum Industry. V. I. Frolov. *The Petroleum Industry*, Nov., Dec., 1927, Nos. 11 and 12, p. 563.

Pre-war royalties were collected by three fundamental methods: (1) Poodage; (2) in kind; (3) conditional payment, coupled with the obligation of a minimum production, and were obligatory even if there were no production.

"Poodage" payment on the Baku Government sections varied between 7/8 and 5.65 cop. per pood. "In kind" was a certain percentage of nett production, with great variations, the maximum being 53 per cent. on plot No. 12 Bibi-Eibat. In Grosny district in kind did not exceed 15 per cent.

"Conditional payment" consisted of an obligatory premium production, all production in excess of this minimum being rated at 20-25 per cent.

At the present time royalties are expressed in poodage and are varied from year to year as follows:—

	Azneft.	Grosny.
1924–25	cop. 2·0 per pood ..	2·0 per pood
1925–26	„ 3·5 „ „ ..	5·0 „ „
1926–27	„ 5·0 „ „ ..	6·9 „ „
1927–28	„ 6·0 „ „ ..	6·9 „ „

Characteristic of the way in which the foregoing royalties have been augmented is the following. The N.K.F. proposed that as the nett profits of Azneft and Grosny had continuously increased, absolutely and relatively, it would be correct to enhance their royalties for 1927–28 from 5 to 6 cop. per pood for the former and from 6·9 to 10 cop. for the latter. After discussion and as the Trans-Caucasian Federation could not make its budget balance, it was decided to increase Azneft royalties by 1 cop. per pood and to leave Grosny without change at 6·9 cop., this decision to remain in force six months, during which period the whole question of royalties to be studied and investigated.

Since nationalisation royalties have been fixed without any basis, increasing year by year, such instability deprives the industry of the possibility of preparing plans five years in advance. Azneft, in planning for five years in 1923–24 calculated their royalty at 2 cop. per pood on 450 mill. poods; now they have to pay 6 cop. or 200 per cent. more, an excess payment over estimate of 18 mill. Rbls.

To return to the old system of 24 years or until "worked out" is too long, as it is not possible to correct any errors, and as the working programme is now accepted for a five years period, it would be most suitable to fix royalties

for the same period. By the end of the fourth year sufficient material will have been collected to make any alteration necessary in the royalties, during the ensuing five years term.

After considering the cost of production, transport from the more distant field to the market and comparing the richer productivity of some sections and fields with the poorness of others, the author recognises the following fundamental circumstances :—

1. Royalty in the oil industry is a rental payment for exploiting oil-bearing lands.

2. This is an income for the landowner, arising from the monopoly of the land.

3. In theory this income should be equivalent to the rent.

4. The rent is an excess of the nett income of the undertaking, which is received in the price of the production after covering all expenditure of production and mean profit.

5. To calculate the rate, as far as land rent is concerned, all expenditure in oil producing and the price of crude oil should be taken into account.

6. The excess of nett income in the oil industry, unifying oil production and oil refining into one whole unit, above the average industrial profit, is not a land rent and in calculating royalties should not be considered.

7. The rent is of two kinds—differential (relative) and absolute. In conformity with these royalties are divided into two similar parts.

8. Differential rent is received from all working sections, excepting the "very worst." Its rate is fixed by the natural qualities of the section (productiveness); these qualities are expressed in variations of production unit cost price (pood, ton). Absolute rent, if it has a place, is the same on all working sections, and its rate is fixed by the height of the price.

9. As represented by the sum of these two items (differential and absolute rent) of which one (differential rent) must vary for different sections, royalties cannot be the same for all working sections.

10. In calculating the rate of rent, profits of an average percentage on the whole industry should be discounted as well as the self-cost of a production unit (exploitation, overhead charges, amortisation).

11. As exploiting sections in the oil industry the districts accepted could be Baku, Grosny, Ural-Emba. The calculation of rents in sections of districts is of interest to the Oil Trusts, but not to the Government, inasmuch as they look upon each Oil Trust as a single ownership unit.

12. "The worst" section, *i.e.* that with whose revenue other sections are compared, is the Ural-Emba district, where the self cost of the production unit is the highest. This defines that Baku and Grosny districts have differential rents.

13. The realisation of the rent is dependent on the price of oil. The price cannot be lower than self cost on the worst district—the Ural-Emba. If it is equal to this self cost, then only Baku and Grosny districts can obtain rent. In so much as the price is higher than the self cost of Emba oil, just so much will be the (absolute) rent for the Ural-Emba district, and to that amount will the rent be increased on the other two districts, where it will compose the sum of the differential and absolute rent.

14. The practical admission, altogether not in accordance with facts, that the oil of various districts is of one and the same value for consumers may be adopted under our conditions, where production and refining of oil are concentrated in one hand—"Government Oil Trusts."

15. In as much as one and the same price is designated franco-place of production in so much may the difference in the geographical position of the district be set aside from consideration.

16. Royalties should be apportioned for a more or less prolonged term, even if for a minimum of five years; if otherwise, working and financial plans lose significance.

17. In reviewing rates of rent, only revealed alterations in the natural qualities of the oil deposits can and should be taken into consideration. To decrease self cost owing to rationalisation increases the profit, and not the rent. T. W.

268. Grosny. Record Depth Installation of 3 in. Deep Well Pump. General News. *The Oil Bulletin*, May 15, 1928, **10**, 11.

A 3 in. deep well pump has been installed at a depth of 1067·3 m. in well No. 3/11 drilled to a depth of 1218·3 m. to the XIV. horizon. It has three valves, with a stroke of 32 in., and delivers 32·5 tons daily. This is the deepest 3 in. pump in Grosny district and probably in Russia. T. W.

269. Gas Source of "Daghestan Fires." General News. *The Oil Bulletin*, May 15, 1928, **10**, 12.

At a distance of 11 klm. N.W. of Derbent and 4 klm. from the Caspian Sea-shore is situated a gas source known under the name of "The Daghestan Fires."

Pospecting work has lately increased the gas producing area from the original 580 sq. metres to 99,880 sq. metres. Of the 54 hand-drilled wells with an average depth of 45 metres, 29 disclosed gas inflows, proving it to be a gas-producing area, meriting further deeper drilling. Five of these wells produced about 32,000 cub. metres daily, which is equivalent to 1,700 poods of oil. A deeper prospecting well, to investigate the capacity of the gas strata, was drilled and five flows of gas were met with between 45 to 80 m., which was utilized through between casing space to an amount of about 14,000 cub. metres per day. At 165 m. a powerful gas fountain was met with, which destroyed the derrick top and threw out earth to a height of 40 m. Upon placing the casing head in position the pressure was found to be 12 atm. This powerful fountain could not be utilized, as plugging continuously formed.

As a result of investigations it must be acknowledged that colossal reserves of gas are hidden in the interior, and that this breaks through by means of crevices and cracks to the open at a tremendous distance from its source. Also, there is no doubt that oil deposits are underlying the gas seams, which is definitely proved, irrespective of other considerations, by the presence of signs of oil in three shallow prospecting wells.

Serious attention should be given to the "Daghestan Fire" area and immediate steps should be taken to put down three wells to a depth of 700–800 metres. At the present time well No. 41 is in exploitation with a daily output equalling 100,000 cub. metres with a pressure of 20 atm.

T. W.

270. Baku. Fountain at Kara-Chkure. General News. *The Oil Bulletin*, May 15, 1928, **10**, 11.

This prospecting fountain yielded during April about 10,000 tons and at the present time 550 tons daily.

As the contour of the oil bed has not yet been defined and also to discover the saturation of the Suralhani sands, found in well No. 2, with signs of oil, Azneft has decided to drill five more wells at Kara-Chkure and one at Ziekh, the first on order being one to the W. of No. 2 well, one to the S. and one to find out the saturation of the Surachani masses.

The oil is of a somewhat different composition to the usual Surachani oil; S.G.—0,8508, tar content 9 per cent., paraffin 4·3 per cent.

In distilling boiling commenced at 74°. Up to 150°—6·1 per cent. gasoline was obtained. From 150° to 300°—39·5 per cent. kerosine, S.G. 0·8159. The residue—mazoot 54·4 per cent., S.G. 0·8818 with freezing at +27°, and tar content 15 per cent. T. W.

271. TESTING TANK CAR VALVES. D. V. Stroop. *The Ref. and Nat. Gas Mfr.*, Vol. 7, No. 1, p. 101.

Tests have been made by the United States Bureau of Standards upon a number of 25 lb. safety valves and 4 in. bottom outlet valves for use on tank cars transporting petroleum products. The valves and bottom outlets to be tested were mounted upon a 2,000 gallon tank suspended by four wire ropes so as to swing on a radius of about 15·2 feet. The tank was arranged to be drawn back and allowed to swing against a stationary tank car loaded with 6,000 gallons of water, the customary spring buffing gear on both tanks being replaced by a solid buffer plate.

Outlet valves were tested mainly with a view to investigating the effect of impacts upon the tightness of the valve. Most valves were tested by giving the tank 15 impacts at a speed of five miles per hour and one additional blow on the end of the outer casting to determine the effect of the breaking of the casting upon the valve. Operative tests were also made and each valve opened and closed several hundred times. The approximate free discharge capacity was determined. Of the valves tested, those with non-metallic closure material proved superior, but this should be expected with short duration tests and may not hold in actual service.

Safety valves were tested for air tightness under pressure and for approximate discharge capacity.

The detailed results of all these tests are available at the U.S. Bureau of Standards, but the main object of this article is to put on record the fact that tests of these types of valve may now be made and to encourage designers to utilise the facilities for testing which are now offered. E. R. B. S.

272. THE RECOVERY OF ACID FROM SLUDGE OIL. *The Petr. Times*, Vol. 19, No. 470, p. 70.

In a paper read by Mr. W. N. Davis, of the Standard Oil Company of California, before the American Petroleum Institute, a method of heating sludge oil under pressure and to relatively high temperatures was described.

By applying a pressure slightly greater than that exerted by steam over acid of the strength being recovered, many sludges may be heated as high as 350° F. for periods up to two hours, thus producing clean acid for subsequent concentration and a tar of low acidity and viscosity which, compared with the original sludge, is an excellent fuel.

The exact temperature, reaction time and dilution of acid to be produced for best results in the hydrolysation depend upon the properties of the sludge being treated. Pressures may vary from about 25 lbs./sq. in. for naphtha sludge to about 65 lbs./sq. in. for fuming acid kerosine sludge.

The first retorts erected were lined with pure 16 lb. lead and operated by a batch method. Results were excellent as regards recovery, but deterioration of equipment was excessive. Better results have been realised in the working of plant as a continuous process, so avoiding one of the most serious drawbacks of the batch method, alternate heating and cooling, which accounted for most of the lead failures.

The latest design of continuous plant has a retort consisting of a steel shell, about 30 feet long by 6 feet in diameter, lined with heavy sheet lead overlaid with acid-proof brick set in acid-resisting cement. Sludge, water and steam in the correct ratio pass together into a mixing device and thence into the

retort or digestor. The acid and tar separate into layers and may be withdrawn independently. It will be seen that by suitably arranging the outlets any desired ratio of acid to tar volume may be maintained. It will be found advantageous to remove the tar as it forms so as to leave a relatively thin layer in the retort. By so doing the tar will be less subject to oxidation and will leave the retort in a more fluid condition, while at the same time there will be more room for acid in the chamber. The effect is to give a long digestion time at high temperature, essential to the production of clean acid.

The tar produced has a low acidity and forms a good refinery fuel, while the acid yield is increased and obtained in a form which, due to its low carbonaceous content, is readily concentrated. E. R. B. S.

273. Improved Process for the Oxidation of Hydrogen Sulphide contained in Industrial Gases to Sulphur. I.G. Farbenindustrie Aktiengesellschaft. E.P. 282,508. Dec. 28, 1927.

The invention provides for a process for the oxidation of hydrogen sulphide contained in industrial gases to sulphur by the use of active carbon or other highly adsorptive material in which the gases on their way to the reaction zone are brought into intimate contact with water or steam giving them a humidity of 80 per cent. or more and maintaining this until the reaction is complete. S. W. C.

274. Recovery of Reaction Products from Gases Treated with Electric Arcs. I.G. Farbenindustrie A.-G. E.P. 288,056. Dec. 12, 1926.

This is a patent of addition to No. 263,859 (Conv. Germany, Dec. 30, 1925), and consists in producing pure hydrogen from hydrogen-methane mixtures, or a gas rich in nitrogen oxides from air, by treatment in an electric arc furnace and separation of the reaction gases into a portion containing hydrogen and acetylene from which acetylene is removed, and a portion containing hydrogen, unconverted methane and small amounts of acetylene, or into two portions containing a high percentage and a small percentage of nitrogen oxides respectively. G. S.

275. Improved Process for the Activation of Carbon or Carbonaceous Material. I.G. Farbenindustrie A.-G. E.P. 289,170, 1928.

The activation of carbon or carbonaceous materials is brought about by causing an activating gas or vapour, *e.g.* steam, to diffuse through a porous membrane into a body of the heated material, the products then diffusing out of the activating chamber. Alternatively, the steam, etc., may be introduced directly into the chamber. The gaseous products may be burned and the heat of combustion used to maintain the temperature of the material undergoing treatment. H. G. S.

276. Manufacture of Metallic Catalysts. I.G. Farbenindustrie A.-G. E.P. 282,410. 1928.

A modification of E.P. 281,218. Hydrogen is used to precipitate metals from their ammoniacal solutions in the manufacture of catalysts, such as nickel, cobalt, copper or a mixture thereof. The ammoniacal solution is preferably treated in an autoclave under pressure either in presence or absence of a suitable carrier and the precipitation may be accelerated by the presence from the beginning of a metal having a catalytic action, *e.g.* nickel, cobalt, copper or the like. Thus a solution containing 119 parts by weight of nickelous chloride, 15 parts of ammonium chloride, 600 parts of water and 250 parts of a concentrated solution of ammonia is mixed with 2 parts of finely subdivided

nickel and 150 parts of kieselguhr, and the whole treated with hydrogen in an autoclave at 100° C. under a pressure of 30 to 35 atm. After some hours, the nickel is completely precipitated in a finely divided state and may be used in the dry or moist condition for the catalytic hydrogenation of organic compounds. H. G. S.

277. Improvements in Carrying Out the Catalytic Oxidation of Organic Compounds in the Gaseous or Vapour State. I.G. Farbenindustrie A.-G. E.P. 272,557. 1928.

Practically pure products may be obtained in the catalytic oxidation of organic compounds in the vapour state by passing the reaction mixture leaving the reaction chamber through suitable solid adsorbent substances prior to separation of the reaction products. Suitable adsorbent materials are those which possess a large superficial area, *e.g.* granulated pumice, eathernware, silica gel, active carbon, etc., and which do not alter their shape by melting or volatilisation at the temperature employed. The treatment is carried out preferably in a continuous manner so that the products first come into contact with partially spent material. In some cases, the adsorbent materials are improved by impregnating them with solutions of salts or bases or by coating them with salts or metallic oxides. Thus commerical naphthalene is catalytically oxidised and the products passed at 260° C. through granulated pumice. The bulk of the product obtained on cooling is perfectly white and contains more than 99 per cent. of phthalic anhydride. H. G. S.

278. Temperature-Measuring Devices. John Liddon Orchard and Cambridge Instrument Co., Ltd. E.P. 287,810. Nov. 11, 1927.

This is a patent of addition to No. 248,301 of Dec. 22, 1924. The instrument embodies five thermo couple strips, which are connected electrically in series, the E.M.F. being approximately five times that of a single strip for equal differences between hot and cold junctions. It is suitable for the measurement of lower temperatures than the normal pyrometer, giving a full scale deflection for a rise of 50° C. The device is applicable to measurements by direct contact in the case of electrically unconductive materials. With conductive materials an insulating film is interposed between the strips and the object under examination. A modification permits of distant use if required, with variable relative positions of the thermo couple strip to suit conditions. It can also be adapted for measurements on concave surfaces. G. S.

279. Water Softening with Barium Salts. C. J. Rodman. *Chem. and Met. Eng.*, 1928, **35**, 221–223.

In treating any boiler feed water the cost of chemicals is no measure of the economy. Commercially, it is found that the use of lime-barium water softeners are particularly adapted to waters impaired by an excessive sulphate content. The use of barium hydrate as a water softening chemical is well known, but recent methods have been devised for using the less expensive barium carbonate with the same efficiency.

The new treatment consists in treating a certain mixture of lime and barium carbonate with steam, thus partially converting a proportion of the barium carbonate to hydroxide. The softening agents are then introduced into the water and the first reaction is the removal of dissolved CO_2, calcium and magnesium bicarbonates and other magnesium salts by the mixed hydroxides. The sulphate radical is then eliminated by precipitation as colloidal barium sulphate, which is coagulated by the sodium chloride present. H. S. G.

280. Improvements in or Relating to Gas Analysis Apparatus. Electroflo Meters Co., London. E.P. 288,510. April 12, 1928.

A gas burette of the Orsat type is fitted with an open, slightly flanged bottom and a two-way tube at the top. The burette is surrounded by a glass jacket open at the top and having a stopper and vent pipe placed in the opening. The bottom of the jacket is also fitted with a bored cork carrying a tube and to this by a flexible tube an aspirator bulb is fitted. The bottom of the gas burette is held away from the tube in the cork by means of a light spring between the annulus of cork and the flange on the burette itself. To one end of the T piece at the top of the burette is attached an absorption bell fitted with steel wool or other similar substance surrounded by a bottle fitted with a cork and breather tube containing the absorbent. The other end of the T piece is connected through a mercury seal to an aspirator bulb connected with the source of gas.

In operation the lower bulb is fitted with water and attached to the empty burette and water jacket. The upper aspirator bulb is then deflated repeatedly until the burette and jacket have been filled with uncontaminated gas from the source. The lower bulb is now emptied by pressing, thus forcing the measured quantity of gas trapped in the burette into the absorbent. When absorption is complete the water is allowed to recede into the bulb till the burette is at atmospheric pressure and the volume noted. There may be any number of absorption pipettes used. A. R. S.

Chemistry of Petroleum.

281. Preparation of Butadiene. S. F. Birch. *Ind. Eng. Chem.*, 1928, **20**, 474.

Butadiene can be readily prepared in the laboratory by distilling "railway hydrocarbon" (the liquid produced when oil gas is compressed), and passing the gases produced into cooled bromine.

After destroying the excess of bromine by means of sulphur dioxide, the solid butadiene tetrabromide is filtered off and crystallised from petroleum ether (b.p. 60–80°). Fractionation of the oily bromides from the filtration yields a further crop of the solid tetrabromide, while the fraction b.p. 165–170°/42 m. consists of the liquid modification mixed with some of solid form. The butadiene is regenerated from the tetrabromide by means of zinc dust and alcohol in the manner described by Thiele (Annalen., 1899, **308**, 337).

An attempt to isolate isoprene from the low boiling fractions of "railway hydrocarbon" was not successful. S. F. B.

282. The Light Oil Residues of Oil Gas Tars from Solar Oil (Gas Oil) and from Crude Naphtha. By Eng. Chem. E. Pyhälä, Helsingfors. *Erdöl und Teer.*, Vol. 4, No. 9, p. 146.

A comparison is made of the light oil residue freed from xylol from light oil gas tar occurring in the pyrogenic decomposition of solar oil with the corresponding fractions from oil gas tar of Romany naphtha, and special consideration given to the decomposition and content of technically useful products.

I.

The light oil residue obtained from solar oil gas tar after distilling off the xylol was fractionated before and after pre-treatment—namely, addition of $HgCl_2$ solution—the colloid formed being removed by filtration, the oil then

being mixed with floridin. The light oil residue was fractionated from a cast iron 20 kgm. boiler with a column, the fraction boiling between 150–170° C. (specific gravity 0·870 at 15° C.) collected and tested. This fraction was deep yellow in colour and the odour was not unlike tetralin. It was distilled from a glass flask with a Glinsky dephlegmator; the principal fraction (150–170° C.) was bright yellow like turpentine, the residue was a very viscous brightly drying varnish, of which about 22 per cent. consisted of solid colophonic resins. The 150–170° C. fraction was rendered water-white on redistillation and resembled turpentine. The above described pre-treatment appeared to cause polymerisation in the original oil due to the action of floridin.

II.

The corresponding fractions of oil gas tar from Romany petroleum were examined likewise. The light oil residue was distilled from a 20 kg. cast iron boiler. It was found to contain some water, but no turpentine oil, varnish or resin. The fraction boiling between 140–150° C. was dark brown owing to the presence of dissolved organic bodies, neutral, and gave no colouration or precipitate with $HgCl_2$. Naphthalene crystallised from the whole fraction at room temperature and crystallised completely at 0° C. The fraction 230–233° C. gave oily but pure white naphthalene. The fraction boiling between 150–170° C., specific gravity 0·8487/20° C., was redistilled after treatment with aqueous $HgCl_2$, then with floridin, and finally distilled from a glass flask.

III.

Similar treatment was applied to Baku petroleum, but three methods of distillation were carried out.

(*a*) Distillation with superheated steam. Fraction to 160° cooled to —10° C., filtered at this temperature. It was found that the naphthalene passed over with the steam as the lower boiling fractions and a light volatile compound and was very soluble in these at room temperature.

(*b*) Distillation from an ordinary glass flask with a Glinsky dephlegmator. Naphthalene separated on strong cooling.

(*c*) Distillation from a cast iron 20 kgm. boiler. Naphthalene crystallised at room temperature, but the fractions boiling between 215–220° C. and 220–230° C. did not solidify absolutely at room temperature. This distillation confirms that a partial cracking distillation of this substance produces two to three times the yield of naphthalene than that obtained by distillation with superheated steam from a glass flask. Since no turpentine oil was present in the Baku petroleum, the xylene distillate residue was tested. The latter contained no turpentine oil.

The following conclusions summarise the effects of different decompositions of light oil residues from oil gas tars from solar oil, Romany and Baku petroleums.

1. The light oil residues obtained from solar oil (gas oil) contain products similar to turpentine oil, varnish-forming oils and neutral resins.

2. These products are not present in the light oil residues arising from Baku petroleum, but reasonable quantities of naphthalene are obtained.

3. The yield of the product named in (1) can be increased by pre-treatment of the corresponding crude substance with floridin, the quality of the turpentine oil substitute being simultaneously improved.

4. The yield of naphthalene from the crude named in (2) can be doubled or tripled by a suitable distillation causing partial decomposition.

5. Examination of the xylene distillate residue shows that the same only contain hydrocarbons like solvent naphtha. W. S. C

283. Binary Azeotropic Mixtures of Ethyl Alcohol with Petroleum Hydrocarbons. Y. Tanaka and T. Kuwata. J. Coll. Eng. Tokyo, 1927, xvii. (5), 117–126.

The azeotropic mixtures formed by ethyl alcohol and hydrocarbons have been investigated and a simple relation found to exist among the boiling points of hydrocarbons or their mixtures and the compositions and the boiling points of the azeotropic mixtures formed by the hydrocarbons or their mixtures and ethyl alcohol.

The relation between hydrocarbon boiling points and the weight percentages of the hydrocarbons in the azeotropic mixtures may be represented by a straight line expressed by the empirical formula—

$$W_H = \frac{160 - T_H}{1{\cdot}15}$$

for paraffins and naphthenes, and

$$W_H = \frac{140 - T_H}{0{\cdot}89}$$

for aromatics, where W_H and T_H are respectively the weight percentage and boiling point of the hydrocarbon in the azeotropic mixture.

The relation between hydrocarbon and azeotropic mixture boiling points may be represented by a hyperbolic curve expressed by a formula—

$$(T_H - T_{AZ})\,(81 - T_{AZ}) = K,$$

where T_{AZ} is the boiling point of the mixture and K a constant nearly equal to 236 for paraffins and naphthenes and nearly 150 for aromatics.

Hydrocarbons or their mixtures boiling below about 45° C. or above about 160° C. can hardly form azeotropic mixtures.

The above formulæ are also applicable to hydrocarbon mixtures having a wide boiling range, provided T_H and T_{AZ} are taken as mean boiling points.

G. S.

284. On the Neutralisation of Oils Rich in Naphthenes and a New Dry-Refining Process. By Eng. Chem. E. T. Leemans, Hague. *Petr. Zeit.*, Vol. 24, No. 13, p. 548.

This research was carried out on an oil which could not be bleached after the usual lime refining process. After the addition of silicate and precipitation of acid tar, the acid value before treatment with lime varied from four to six. Both the unrefined and refined oils were inclined to emulsion formation and were malodorous.

In the neutralisation of the crude oil, the carbonates had to be completely separated, since naphthenic acids are weaker than carbonic acid. Similarly with caustic soda. About 60 per cent. of the naphthenic acids were bound in the crude oil and so much of these acids distilled over with the oil as to cause hydrolysis of the sodium salt. The neutralisation with dissolved lime was complete. The pulverised lime was allowed to intersperse with the oil in a preheater, the temperature varying from 40°–100° C., the oil being kept in motion by an air-blast; 100 c.c. oil were distilled down to 10 c.c., the distillate titrated in a neutral quantity of alcohol and ether with normal lye. If cracking does not occur in the distillation, the calcium naphthenate is undecomposed and passes into the asphalt. The acid value of the naphthenic acid separated from the lubricating oil amounted to 56·88. The crude oil contained about 10 per cent. of naphthenic acid.

In the case of lubricating oils all the distillates contained unremovable sulphonic acids arising from the acid tar dissolved in the oil, which was

unaffected by soda lye. A means of removing this acid tar was the application of a dilute solution of water-glass. Thirty litres of a solution containing a proportion of 9 litres water and 1 litre of water-glass (40° Bé) were added to each 6,000 litres of oil, and after further stirring 10 litres of this solution were added. The acid tar sank immediately to the bottom of the vessel, the particles of sulphonic acid were neutralised at the surface of contact with the water-glass whilst the silicic acid formed prevented the sodium sulphonate from passing into the oil.

The three factors which are of importance in this process are a slight neutralisation, an adhesive effect and an adsorption of hitherto harmful oil constituents by the silicic acid gel formed. The drained acid tar is still found to contain unchanged water-glass since it turns blue in air. The oil is then heated in a shallow beaker to 40°–60° C., and the acid tar removed after a short time.

In the dry refining process the oil is pumped into the bleacher, heated to 100° C. and kept in motion by means of an air-blast. One to two per cent. of dissolved lime powder is applied and the oil is passed into a clear vessel, thence to the filter-press. Calcium silicate separated in the clear vessel. The acidified oil appeared to be clear, but it still contained particles of acid tar enveloped with water-glass, which formed large lumps in the neutralisation. The lubricating oil so refined is orange-yellow and could not be bleached.

In the attempts to refine oil which had no lime treatment in the above manner, it was found to contain 0·38 per cent. ash after refining, whilst lime-treated oil contained only 0·0095 per cent. ash. The latter did not emulsify with water, but this property predominated strongly in the former. The oil had a good smell after the above refining and the freezing point was lowered from +2° C. to —6° C. These last two properties are chiefly due to adsorption by the silicic acid gel formed. W. S. C.

285. THE PHENOLS FROM NATURAL PETROLEUM. Y. Tanaka and R. Kobayashi. J. Coll. Eng. Tokyo, 1927, XVII. (6), 127–133.

The paper describes investigations on identification of the phenolic compounds occurring in Japanese petroleum.

It was previously suggested that a marked amount of phenolic compounds existed in the naphthenic acid fractions boiling below 150° C. (8·9–9·0 mm.), and the authors found it convenient to extract these compounds from crude naphthenic acids from kerosine distillate.

The kerosine distillate (49·2–42° Bé) from Akita petroleum was passed through a concentrated caustic soda solution, acid substances being separated with dilute sulphuric acid. It has an acid value of 148·4, using phenolphthalein as an indicator.

The acid mixture was just neutralised by adding a calculated amount of potassium hydroxide and the resulting soap solution treated with ethyl ether to extract phenolic substances.

The phenols were further purified by a repetition of the above procedure and by converting them into potassium phenolates by repeatedly extracting the latter with petroleum ether to remove hydrocarbon oil. The phenolate solution was heated to expel the ether and decomposed by hydrochloric acid. After washing and dehydration the resulting phenols were a deep brown, mobile oil with a strong smell like cresol.

About 260 grams of purified phenolic compounds were obtained, giving a pale yellow oil with a strong odour, sp. gr. $\left(d\frac{20}{20}\right)$ 1·0315 and refractive index

$\left(n\frac{20}{D}\right)$ 1·5402. Its properties point to a mixture of the mono-phenols.

By repeated fractionations of the mixed phenols it was found that the boiling points, etc., of the main fractions closely resembled those of cresols, xylenols and ethylphenols.

The chief constituents of the phenolic compounds are cresols, notable amounts of p-cresol, m-cresol, o-cresol, 1, 3, 4-m-xylenol and small quantities of 1, 2, 3-o-xylenol, diethyl phenol and triethyl phenol being found. G. S.

286. Phenols in Petroleum Distillates. Le Roy, G. Stoup, and Robert D. Snow. *Ind. Eng. Chem.*, 1928, **20**, 359–364.

Spent alkaline solutions from the refining of both cracked and straight-run petroleum distillates have been found to contain small quantities of phenols. Although these only constitute a small percentage of the distillate treated, they collect in the waste liquors, as a result of which, their disposal becomes a considerable problem. The two methods at present employed are (1) neutralisation of the waste liquor by acid-sludge and burning of the oil which separates, and (2) treatment of the spent alkali with engine exhaust gases thereby converting the caustic soda into sodium carbonate and liberating the phenols which are burnt. The purpose of this investigation was to find a better use for the phenols than as fuel. The relatively large quantity present in cracked distillates indicates that they are largely produced by cracking, although they also exist in the crude oil itself. The phenols isolated were mainly composed of the high boiling members; little or no phenol was found, although cresols were present. By treating selected lower-boiling distillates, it was found that phenols having a composition approaching that of commercial cresylic acid could be isolated. The yield was too small to make the separation practicable commercially. A comparison is made of the phenols isolated with the low-temperature tar phenols. S. F. B.

287. Synthesis of Alcohols Higher than Methanol from Carbon Monoxide and Hydrogen. K. Frolich and W. K. Lewis. *Ind. Eng. Chem.*, 1928, **20**, 354–359.

The synthesis of alcohols higher than methanol from carbon monoxide and hydrogen at elevated temperatures under high pressure has been studied. Two types of catalysts—(*a*) iron impregnated with alkali and (*b*) mixtures of metallic oxides—were employed. The composition of the former was varied considerably. In general it was found that with this type of catalyst it is very difficult to suppress side reactions leading to the formation of carbon dioxide, methane, water and free carbon. The formation of carbon dioxide is probably unavoidable owing to the type of reactions involved, but it is thought that the high temperature necessary for the main reaction is responsible for most of the loss as it was found that the higher the temperature employed, the greater was the loss. The liquid product usually separated into two layers—one containing a large proportion of water and the other, oily in character, consisting of a complex mixture of the lower aliphatic oxygenated compounds and hydrocarbons of various boiling points. Considerable quantities of higher alcohols were obtained using the mixed-oxide catalyst together with methanol, water and gaseous products. The loss of carbon dioxide in side reactions was high in both series of experiments, although slightly higher when using the iron-alkali catalysts. By the use of higher pressures and more reactive catalysts of the mixed-oxide type, it is hoped to lessen the tendency for side reactions to occur. S. F. B.

288. THERMAL DECOMPOSITION OF ORGANIC SULPHUR COMPOUNDS. W. F. Faragher, J. C. Morell and S. Comay. *Ind. Eng. Chem.*, 1928, **20**, 527–532.

This investigation was undertaken with the object of studying the thermal decomposition of organic sulphur compounds under conditions similar to those met with in cracking. The sulphur compounds employed were selected as representative of the types likely to be present in petroleum, *i.e.* mercaptans, alkyl sulphides, alkyl disulphides, thiophene and elementary sulphur. Preliminary experiments showed that the higher disulphides decompose during distillation at atmospheric pressure giving hydrogen sulphide, mercaptan and a tarry residue. The experiments under cracking conditions were carried out by "flashing" the sulphur compounds and passing the vapour produced through a reaction tube heated by means of an electric furnace to an average temperature of 496° C. Approximately 0·5 c.c. of sulphur compound was vaporised per minute. The vapours leaving the furnace were passed through a condenser to separate liquid products and then, after scrubbing by sodium hydroxide solution, collected over water. The products were analysed by the methods described by Faragher, Morell and Monroe (*Ind. Eng. Chem.*, 1927, **19**, 1281). The results obtained may be summarised as follows: Disulphides, as represented by ethyl, *n*-propyl, *n*-butyl and *iso*-amyl disulphides, decompose to give hydrogen sulphide, sulphur, mercaptans, thiophene derivatives and gaseous products belonging to the methane and ethylene series. A pitchy residue was also left. Alkyl sulphides, represented by ethyl and *iso*-amyl sulphide gave hydrogen sulphide, mercaptans and gaseous paraffins and olefines. The only mercaptan tried, *iso*-amyl mercaptan, gave large quantities of hydrogen sulphide, sulphur, hydrocarbons and pitch. No sulphides or thiophenes could be detected. Thiophene was tested in naphtha solution only; no decomposition could be detected between 496° and 871° C. Free sulphur also tested in naphtha solution, gave hydrogen sulphide at 496° C, although traces of this substance could be detected as low as 316° C. Experiments carried out on *iso*-amyl disulphide in naphtha solution indicated that at comparatively high velocities (2 c.c. of 2–3 per cent. solution per minute), the percentage of free sulphur produced is higher, but that of mercaptans is lower. With the exception of thiophene all the substances tested in naphtha solution gave hydrogen sulphide as the chief product. Experiments were also carried out on *n*-butyl and *iso*-amyl disulphide in Grosneft gas oil solution under liquid-phase cracking conditions. The solutions were cracked at 437° C. under 17 atmospheres and the pressure distillates and N.E.P. gasolines from them, analysed. It was found that traces of hydrogen sulphide, free sulphur and substances reacting like thiophene were present in both the pressure distillate and the gasoline and that no unchanged disulphide remained.

The stability of thiophene suggests a reason why certain crude oils or fractions, while high in sulphur content, cause less corrosion than others having much lower sulphur content. Thus Smackover crude contains 2·5 per cent. sulphur, but shows less corrosion during cracking than Panhandle crude containing 0·6 per cent. S. F. B.

289. IMPROVEMENTS IN AND CONNECTED WITH THE MANUFACTURE AND PRODUCTION OF UNSATURATED HYDROCARBONS. I.G. Farbenindustrie A.-G. E.P. 290,322. 1928.

The production of unsaturated hydrocarbons, particularly acetylene, is effected by spraying liquid hydrocarbons into a combustible gas and heating the mixture to a high temperature by burning part of the mixture with oxygen or gases rich in oxygen. The combustion is so effected that a disc-like flame is produced. H. G. S.

290. Synthesis of Higher Hydrocarbons from Water Gas. D. F. Smith, J. D. Davis and D. A. Reynolds. *Ind. Chem. Eng.*, 1928, **20**, 462–4.

Various catalysts were tested qualitatively to determine their efficiency in converting water gas to liquid hydrocarbons at 300° C. under atmospheric pressure. An iron catalyst prepared by the reduction of artificial magnetite and promoted with aluminium and potassium oxides and a cobalt-chromate catalyst were inactive. Cobalt-copper-chromium oxide, cobalt-copper and cobalt-copper-uranium oxide were found to possess the required property, a deposit resembling white vaseline being formed in the case of the last mentioned catalyst, as well as oily and watery substances. Quantitative tests were finally made with a catalyst prepared by precipitating with caustic soda a solution containing cobalt and copper nitrates and manganous chloride and reducing the washed and dried precipitate at 300° to 350° C. Water gas was passed at 200 c.c. per minute over this catalyst, the products being taken through a receiver cooled in liquid air. The oil yield attained a maximum at 275° C., whilst with rising temperature, the contraction in volume, and yields of water and carbon dioxide slowly increased. The hydrocarbons consisted of both saturated and unsaturated products ranging from ethylene and ethane to hydrocarbons which are liquid at ordinary temperatures, but there was no evidence of the presence of very heavy molecules. It is calculated that 1,400 cubic feet of water gas will produce, by this method, about one gallon of oil. H. G. S.

291. A Process for the Manufacture and Production of Hydrocarbons. I.G. Farbenindustrie. E.P. 270,705. 1928.

Liquid hydrocarbons, sometimes associated with those in a gaseous or solid form, are prepared by heating metal carbonyls in the presence of a smaller amount of hydrogen than is required for the production of methane. Mixed carbonyls may be employed, e.g. the carbonyls of iron, cobalt, nickel or molybdenum, and instead of hydrogen, gases containing hydrogen and/or steam or carbon monoxide may be used. The reactions may be influenced by the addition of finely divided copper or alkali metal hydroxides or carbonates. As an example, a mixture of 1 kilogram of iron carbonyl vapour and 400 litres of hydrogen is passed into a furnace heated externally to 240° C. The resulting fine iron powder is separated in dust chambers and the issuing gas passed through active silica from which a readily volatilizable liquid fuel is recovered by steaming. A considerable quantity of less volatile hydrocarbons may be recovered from the iron powder. H. G. S.

Crude Oil.

292. The Breaking of Crude Oil Emulsions by Chemical Means. By Dr. Eng. T. Kuczynski, Lemberg. *Pet. Zeit.*, Vol. xxiv., No. 9, p. 398.

The compounds added to natural crude oil emulsions to effect breaking are classified into two groups: (1) *those soluble in the crude oil*, (2) *those insoluble in the crude oil*. To (1) belong phenol and naphthenic acids which are utilised in the case of Boryslaw-Tustanowice oils. The mechanism of the effect of phenol is explained. Phenol is partially soluble in both phases of the emulsion, increasing the mutual solubility of the crude oil and of the water, and decreasing the surface tension. Asphaltic substances (resins, etc.) collect on the surfaces of both phases and form a protective colloid of the natural emulsion, and the stabilising film consumes the water drops and causes a rapid breaking

of the emulsion. The property of phenol in dissolving aliphatic hydrocarbons is limited, but asphalts, resins, etc., dissolve easily. Group (2) includes tret-o-lite and very dilute alkaline soap solutions which have a small surface tension against the crude oil.

The stability of colloids depends on the sign of the charge on the water-drops. This was found to be negative for American natural crude oil emulsions and for Californian emulsions. With Boryslaw-Tustanowice emulsions the charge was determined either by cataphoresis or by a specially designed ultramicroscope. By means of a very bright light in the macroscopic experiments and by utilising a low percentage (calculated on the crude) of a reddish-brown emulsion, the observations could be carried out further by the ultramicroscope by dilution with light oils.

The observations showed in macroscopic researches with a potential drop of 12–25 volts/cm. that the emulsion was darker at the cathode and brighter at the anode, and the water-drops went to the anode. The reverse occurred with a stronger illumination. This is due to the change in the ionic concentrations in the region of the electrodes. Cataphoresis experiments with macroscopic observations of seven samples of natural crude oil emulsions showed that the water-drops were + vely charged, and this could be completely confirmed by the ultramicroscope.

Dr. Kuczynski carried out preliminary experiments on artificial emulsions of water in unrefined yet heavy transparent spindle oils containing at least traces of all those substances present in the crude, and capable of influencing the sign of the charge. Pure water and various anions and cations—viz. ($NaCl$, $CaCl_2$, $FeCl_2$, $ThCl_4$—Na_2SO_4, K_2FeCy_4, K_4FeCy_6) were utilised in the production of emulsions. The water-drops were shown to be + vely charged in all these emulsions. The addition of acids and bases caused the water-drops to pass to the cathode, but N. and N/10 NaOH behaved abnormally. With highly refined transformer oil emulsion the water-drops went to the cathode.

The results of these experiments is explained by Cohen's rule that the medium with the higher dielectric constant is charged + vely against that with a lower dielectric constant—*i.e.*, water and aqueous solutions are always + vely charged against pure oil (pure hydrocarbons); also Ellis' rule that a movement of oil drops in oil-in-water emulsions is always to the anode. However, from a modern point of view, the water-drops can be regarded as having an "electrically double layer" of pure hydrocarbons, the outer surface – vely and the inner surface + vely charged. Impurities in unrefined oil form a protective film, acid and water-drops being + ve against the unrefined oil and – ve against the alkali.

The writer produced artificially crude oil emulsions using fresh H_2O, and saturated $Ca(OH)_2$ and N.NaOH solutions respectively. In the first case the charge on the water-drops was + ve, and in the latter – ve. These results were confirmed ultramicroscopically by dilution with benzine. With a Boryslaw-Tustanowice crude oil emulsion the water-drops were found to be + vely charged, thus the salt water separated from it reacted acid. The water separated from Texas and Californian emulsions reacted alkaline.

The above discoveries show (1) that the emulsions arising from almost neutral water are barely stable since the charge on the water-drops lies in the region of the isoelectric point. (2) When corresponding quantities of an acid and of an alkaline emulsion are brought together the solidity of the emulsion is strongly decreased and precipitation occurs eventually. (3) When the acid emulsion formed is shaken with alkaline-reacting water, the emulsion breaks on account of the difference in the sign of the charge on the limiting surface, since the alkaline – vely charged water solution must

consume the acid reacting + vely charged water-drops. This explains the extraction of emulsions with NaOH in many Boryslaw-Tustanswice samples.

The breaking of emulsions is effected more quickly by these methods than by ordinary heating. The second statement was confirmed by using natural acid, and artificial alkaline water-in-oil emulsions; these were added to water, mixed, and the reaction performed macroscopically and ultramicroscopically. The solidity of the emulsion depends on the protective effect of asphalt as well as the electric charge; consequently, for separation purposes, the aqueous solution must contain bodies that suppress the protective effect of asphaltic substances. The application of ordinary alkaline solutions is limited in the separation of crude oil emulsions.

The effect of the extraction of natural emulsions with ordinary fresh water still remains doubtful. Here we are dealing with a greater surface contact of emulsion with water and with a further formation of emulsion which is unstable. The isoelectric point of the drops is then quickly attained, and the emulsion becomes more unstable. This also appears to explain the mechanism of the effect of water, alkaline solutions, alkaline soap solutions, starches, pastes, and similar water-soluble colloids.

The separation of emulsions by refining with strong acid is still recommended in the patent literature. The acid tar produced during refining with strong sulphuric acid is extracted with water to remove excess acid, and is utilised in a fresh condition for the breaking of emulsions, dissolving colloidally in the crude oil. The sign of the particles estimated by treating a gas oil with H_2SO_4 and the cataphoresis experiment employed showed that the charge was negative. The separated acid was also – ve to the crude oil and was suitable for the deposition of + ve by charged water drops of natural emulsions.

Emulsions of clay in crude oil were found to be broken by heating the aqueous emulsion periodically under 6 atmospheres pressure and adding ·2% phenol. The charge was determined by treating a natural clay emulsion (free from water) and after strong dilution with benzine, in the ultramicroscope when the clay went to the anode and was – ve to the oil. However, when another stronger aqueous clay emulsion was submitted for a longer time to cataphoresis water separated at the cathode, and clay and sand at the anode. The blending of clay and water emulsions appears to cause mutual coagulation, and this "oil-soluble" and oil-soaked clay forms a protective film for the water-drops.

The fact that clay and sand are – ve to the crude oil is explained best by the effect of filtration through sand or other porous substances, when the latter withdraw asphaltic substances from the crude oil at the beginning of the reaction, thus causing a weakening of the protective film on the water drops.

In conclusion, the Texas and Californian emulsions are the reverse of Boryslaw-Tustanowice emulsions, in which the water drops are + vely charged. The water and aqueous solutions are always found to be + vely charged against pure, highly-refined oils, whilst with unrefined crude oil the acid is + ve, the alkaline – ve. The deposition of acids, mud, clay and sand is charged – vely against the crude oil.

References of the works of German and American patents and current literature dealing with this article are cited in detail. W. S. C.

293. Process for Treating Hydrocarbon Oil. W. M. Cross. U.S.P. 1,667,883. May 1st, 1928.

The process is an arrangement of plant whereby crude oil is distilled in a fire still. The distillate is passed through dephlegmators and the residue is

run down for fuel. The dephlegmator towers separate the vapours into gasoline, kerosine and gas oil. The gasoline is treated and again distilled, giving spirit and kerosine residue which returns to the kerosine stock tank. The gas oil and, if desired, the kerosine, is passed through a cracking coil and reaction chamber, the gas is separated and the synthetic crude collected and charged along with the raw crude to the crude oil fire still. Cracking is conducted at a pressure above 500 lb. and substantially in the liquid phase. In this manner a continuous cycle is maintained for obtaining the maximum yields of lighter fractions from the crude oil.

R. P.

Natural Gas.

294. Theory and Practice of Fractionation. L. S. Gregory. *Oil and Gas J.*, May 24, 1928, 93.

Natural gas is mainly composed of the paraffin hydrocarbons, ranging from methane to octane, and natural gasoline contains from a trace to 5 per cent. of propane, 0 per cent. to 40 per cent. butane, 15 to 40 per cent. pentane, and the remainder is composed of hexane, heptane and octane. The natural gasoline manufacturer of to-day is confronted with the problem of making a gasoline containing all the pentane and hexane possible, with as much butane as marketing specifications allow and a minimum of propane.

The oldest method of separation, weathering or simple distillation, lived a long life because the manufacturer was interested only in reducing vapour pressure and increasing recovery, with slight regard or knowledge for what was lost.

If a still is fitted with a plain column and a mixture of butane and pentane containing 20 per cent. of butane distilled, the vapours evolved will contain nearly 40 per cent. of butane. By redistillation a further separation may be obtained, but this method involves a number of distillations and great heat consumption. If an analyser is added to the column and the vapours partially condensed, improved separation takes place without additional consumption of heat. The substitution of a plate or packed column improves the process still further. The packed column is filled with rings, steel shavings or other material and has the advantages of low intitial cost. Its disadvantages are sensitivity to slight variations in conditions and the possiblity of channelling, giving poor contact between the descending liquid and ascending vapour.

The plate column, perforated or with bubble caps, has the advantages of ease of control; minor variations of conditions are taken up by the liquid on each plate, and better separation per unit distance of vertical height with good contact between vapour and condensate. The column has a concentration gradient from plate to plate, and the fractionation may be carried on until the butane content in the still is practically nil, whilst the vapours drawn off are practically pure butane. This method is known as batch fractionation and is only used where a special cut is desired.

In common practice continuous distillation is used. The feed is introduced on a plate having a similar composition and the product is withdrawn from the still continuously. For the smooth operation of the plant, the feed should be maintained constant in volume and composition, as variations disturb the gradient established in the column and hinder proper separation. The pressure and temperature of the still should be maintained constant, as these factors determine the volatility of the product. Reflux should be ample and fairly constant. If the volume is too much the column will flood, whereas if it is too small an appreciable amount of the higher boiling components will be carried over.

Fractionation has been proved to be economical, and there are four methods which possess the most important modifications in the fractionation of natural gasoline. Fractionation of gasoline and gas, fractionation of rich oil, fractionation of rich oil and gas, and fractionation of the gasoline. In the first three methods the feed is the raw gasoline diluted with gas or mineral seal oil; in the fourth the feed is the raw gasoline from a compression, absorption or charcoal plant.

In the fractionation of gasoline and gas the hot compressed field gas is introduced into the middle of the column and the finished product is drawn from the still, the still being heated by a steam coil and the analyser cooled with water.

The apparatus for the fractionation of rich oil consists of the still and plate column. There is a water coil above the top plate, but no analyser, and denuded oil is pumped into the top of the column to form the reflux. Efficient use of this type of plant is said to do away with the necessity for a recompressor. Hot, rich oil containing the gasoline is withdrawn from the still and pumped to another still for recovery of the spirit.

For the fractionation of rich oil and gas the compressed field gas is introduced into the column half way up and denuded oil is injected at the top of the column, forming an absorbent and refluxing medium. The hot, rich oil containing only the gasoline desired is withdrawn from the still and passed to the oil still. The column thus performs the double function of absorber and fractionator.

For the fractionation of gasoline the same apparatus is used as for the fractionation of gasoline and gas.

Fractionation of the gasoline and gas, of the rich oil and of the rich oil and gas is generally only applicable to cases where there is a rich field gas and a desire for only a narrow range of products. Fractionation of the gasoline may be applied to a plant working on very lean or very rich gas, and a wide range of products may be had.

Fractionation may be conducted under high or low pressure. The former has the advantages that the reflux temperature is generally more easily controlled and it does away with the possibility of the column freezing when a damp feed is employed.

By the use of fractionation a gasoline may be made which is suitable for aviation, or if a special cut is desired it may easily be obtained R. P.

295. A Process of Obtaining Volatile Hydrocarbons such as Gasoline Benzene or Other Hydrocarbon Oils Contained in Natural Gas, Coal Gas, Water Gas, Air or other Gaseous Mixtures and Apparatus therefor. Masatosi Okochi. E.P. 282,541. Dec. 29, 1927.

The invention provides for a process of obtaining volatile hydrocarbons contained in gaseous mixtures, such as natural gas and the like, by passing the gaseous mixture through towers in contact with solid absorbing or adsorbing material at low temperature under ordinary or high pressure in which the absorbed volatile products are driven off by heat, allowing the compressed gas to expand suddenly on admission to the absorption towers, whereby it attains a low temperature. The volatile hydrocarbons driven off from the absorbing material are condensed by the cooling action due to the sudden expansion of another part of the compressed gaseous mixture, this cooling action being assisted by the evaporation of the volatile hydrocarbons condensed from the expanded gaseous mixture.

The apparatus comprises absorption towers, a compressor for the gaseous mixture, expansion valves to supply low temperature gaseous mixture to the towers, low temperature coolers for condensing the volatile hydrocarbons

driven off from the towers, and compression for supplying additional gaseous mixture into the towers under pressure. The low temperature coolers are supplied with expansion valves, pipes for supplying the expanded gaseous mixture from the coolers to the towers and pipes for supplying additonal gaseous mixture to the tower S. W. C.

Motor Spirit.

296. Careful Fractionation Can Reduce Costs of Treating Cracked Gasoline. H. P. Rue. *Nat. Petr. News*, April 25, 1928.

This paper gives the results of two years' investigation on the possibilities of efficient fractionation. The method of treating raw cracked gasoline with acid and alkali followed by a steam distillation may still be considered standard practice. During the last few years great strides have been made in the development of equipment for the fractionation of light products and bubble towers are largely used, but little attention has been paid to the possibility of using this distillation plant to reduce the amount of chemical treatment necessary.

The principal impurities in raw cracked spirit are colour, sulphur compounds and gum-forming substances, and the fundamental principle of this investigation is that these impurities are not distributed evenly through the boiling range of all gasolines and in some cases at least appear to predominate in certain fractions.

A 13 plate bubble tower was used in the experimental work. Pressure distillate and straight-run naphthas of different types were charged into a pipe still at the rate of 20 gallons per hour. The mixture of vapour and the liquid from the still was flashed in an evaporator and the vapour was passed into the bottom of the tower. Products could be drawn from any desired plate and passed through cooling coils. Vapour from the top of the tower passed through a condenser.

When operating on straight-run distillate, it was possible to take most of the product overhead leaving practically all the coloured compounds to be drawn from the side of the tower in the liquid state. This fraction could then be treated with acid and soda and added to the overhead distillate to give finished gasoline.

With cracked pressure-still spirit, the first 30 to 40 per cent. came over stable and of good colour, but the balance of the product required treatment for colour.

An apparent segregation of the sulphur compounds was found, at least as far as usual corrosion and doctor tests revealed. With one cracked distillate which yielded 70 per cent. of 437° F. end-point spirit, it was found that the overhead distillate was corrosive and sour, but the liquid product from the tower was non-corrosive and sweet. If the total yield of spirit were taken off in the vapour phase it was corrosive and sour to doctor test, but if the temperatures in the tower were controlled so that part was taken over in the vapour phase and part taken off in the liquid phase and later combined, the total was a non-corrosive and sweet gasoline, which had good colour stability even after six months exposure to direct sunlight.

With a different pressure distillate both the vapour phase and liquid phase products were non-corrosive, but the vapour phase spirit was always sour. By controlling the temperatures and the amounts drawn off in three different streams, the doctor sour portion was reduced to 8 per cent.

The work on fractionation gave some interesting points on the gum content of various fractions. In one sample of cracked distillate the 30 to 40 per cent. fraction formed, on standing in storage, many times the amount of gum found in all the other fractions combined. The 10 to 20 per cent. fractions did not

contain any gum, but it is not always the same fractions in different cracked gasolines which contain the greatest amount of gum forming compounds. In one run, when three streams were drawn from the tower, it was found that the middle stream was usually greater in gum content than the other two combined, and by controlling the temperature and amount of each stream it was possible to vary the gum content at will. R. P.

297. Fractionation in Gasoline Treatment. H. P. Rue. *Oil and Gas J.*, 1928, **26** (49), 142–143.

The subject of utilising distillation equipment to reduce treatment and thus reduce costs has received little attention. The chief reasons for subjecting gasoline to treatment before sale as motor fuel are colour improvement, removal of corrosive or objectionable sulphur compounds and gum-forming constituents. These impurities are not uniformly divided through the boiling ranges of gasoline and it is possible by careful fractionation to concentrate each impurity in a small portion of the product, the major portion being marketable as it leaves the condenser.

Attention was drawn to this during the distillation of a cracked distillate. The first few fractions were water white, the higher boiling ones being yellow. On re-distillation of the latter more water white gasoline was obtained.

A bubble tower can be used to fractionate gasoline and by a study of the streams a control system can be applied to produce the greater part in marketable condition. A 13-plate semi-commercial bubble tower was used, pressure distillates and straight-run naphthas being charged to a pipe still at a rate of about 20 gallons per hour.

The vapour and liquid from the pipe still was flashed in an evaporator, from which the vapour passed to the bottom of the tower. Streams could be drawn from any plate in the liquid state and passed through cooling coils, the vapour from the top of the tower passing through a condenser.

It is easy to obtain most of the gasoline in a water white condition. When operating on a straight distillate from a high-grade crude most of the product can be taken overhead as vapour, leaving the coloured compounds to be withdrawn from the side of the tower. The coloured product could be treated with acid and alkali and added to the overhead distillate. When using pressure-still distillates, the first 30 to 40 per cent. would have good colour, the remainder requiring treatment, but a more efficient tower would probably give a larger proportion of water-white products.

With a pressure distillate yielding 70 per cent. of 437° F. end-point gasoline, the vapour phase product was corrosive and sour to doctor, the liquid phase product being non-corrosive and sweet.

The laboratory gum test usually shows that the heavier the stream the greater the gum, but this is not always true after standing in storage. In one case after standing, the 30 or 40 per cent. fraction contained more gum than all the other fractions. After standing a year in direct sunlight the 10 and 20 per cent. fractions contained no gum.

In one case, in which three streams were taken from the tower, the gum content of the middle stream was more than that of the other two combined, which indicates that polymerisation does take place in a bubble tower and can be controlled to a certain extent.

Sulphuric acid treatment will remove some sulphur compounds, but its effect on pure sulphur is negative. Unless temperature and contact time are closely controlled excessive losses are caused. In pressure distillate treatment the losses appear to be in the light ends, the loss in the light ends of one gasoline being about 17 per cent., although the total loss was 13 per cent., indicating polymerisation of some of the light fractions.

It is suggested that it is best to separate gasoline distillate into fractions, each being treated according to its requirements. G. S.

298. Ethyl Petrol. W. A. Whatmough. *Automobile Engineer*, 1928, **18**, 241, p. 172.

The composition of "ethyl fluid" is as follows:—

Lead tetra ethyl	54·6% by volume.
Ethylene dibromide	36·4% "
Monochlor. naphthalene	9·0% "
Soudan IV. (red dye)	·01% "

"Ethyl petrol" as sold in Great Britain for road use contains 6 c.c. of "ethyl fluid" per gallon of No. 1 petrol, thus ethyl petrol contains:—

1 part of lead in 980 parts by weight = ·102% or 3·396 grams lead per gallon.

Ethyl petrol for racing cars contains 11 c.c. ethyl fluid per gallon. Aviation Ethyl petrol contains 15 c.c. ethyl fluid per gallon.

6 c.c. ethyl fluid per gallon raises the H.U.C.R. of that petrol	0·8
18 c.c. " " " " " " "	1·7

On light loads and at cruising speeds most of the lead is ejected from the engine as very finely divided lead bromide; on heavier loads the deposits remain in the engine. Mica cores of sparking plugs tend to become impregnated with lead deposits.

Pure lead tetraethyl is a powerful poison, the symptoms being lowered blood pressure, fall in body temperature, weakened pulse, insomnia, loss of weight, nausea, tremor, delirium tremens. The following symptoms of ordinary lead poisoning are absent, the lead line on the gums, stomachic cramp and wrist drop.

Four years experience shows that the rate of elimination of lead proceeds in the human body sufficiently fast to prevent its accumulation in garage employees. On the road the risks are that the lead in exhaust fumes is water soluble, and that accumulations of lead in the exhaust system during idling are blown out on speeding up. W. N. H.

299. Crude Benzol as Motor Fuel. *Petr. Times*, xix., 489-496.

The fact that crude or imperfectly refined benzol deposits a gummy residue on standing and that in some cases the distillate from this gummy benzol is more unstable than the crude benzol has led to the investigation of this gum formation.

The present views are that gum is produced by oxidation of the unsaturated or at least some class of unsaturated bodies present in the benzol. However the benzol can be stabilized by the addition of certain stabilizers, such as phenol or tricresol.

The amount of the latter required for a benzol containing 5–10 per cent. of unsaturateds is as follows:—

Copper container	·001%—·02%
Iron, tinned iron or steel container ..	·005%—·05%
Glass container	·02 %—·1 %

Cracked gasolines can also be stabilized in this way, the cost being small and the increase in colour only slight. A. R. S.

300. Process of Refining Oils. A. Schwarz, Assr. to Petroleum Sand Products Corpn. U.S.P. 1,656,900. Jan. 24, 1928.

The invention provides a process of refining gasoline containing olefines and diolefines which comprises agitation with a mixture or emulsion of sulphuric acid and saturated hydrocarbon oil of higher boiling point than gasoline; the amount of sulphuric acid in the mixture being such as to remove only the diolefines, distilling the resultant mixture of gasoline and saturated hydrocarbon oil and separately condensing and collecting the refined gasoline and the saturated hydrocarbon oil. S. W. C.

Kerosine.

301. Alumina Gel as a Desulphurising Agent in Petroleum Refining. J. K. Chowdhury and R. C. Bagchi. *J. Indian Chem. Soc.*, 1928, v. (1). 111-125.

Sulphur in the oil has been estimated by the lamp method, suitably modified to give concordant results.

Optimum conditions for activation of alumina gel, i.e., roasting temperature and moisture content, have been determined, and the influence of other factors, such as concentration and nature of the sulphur compounds in the oil, quantity and size of particles of the gel and treatment temperature, on adsorption has also been ascertained. The adsorption isotherm of Freundlich has been found satisfactory.

Most of the sulphur compounds have been satisfactorily removed by oxidation with atmospheric air, using alumina gel as a catalyser and filtration. This is an improvement on the present process of oxidation with sodium hypochlorite on a large scale.

It has also been found that intimate mixtures of alumina gel with other adsorbents, such as activated carbon or silica gel, and particularly the latter, mutually promote the adsorption to a large extent. This is similar to the action of promoters used in preparation of catalysts. G. S.

302. Treatment of Hydrocarbons. W. Hess. U.S.P. 1,668,275. May 1, 1928.

An improvement of the Edeleanu process of refining mineral oils in which losses of sulphur dioxide from the treating system are avoided by previously treating the oil with gaseous sulphur dioxide until about ½ per cent. has been absorbed and thereafter subjecting the treated oil to the action of liquid sulphur dioxide. The oils for treatment with sulphur dioxide are allowed to trickle over a tower filled with Raschig rings at the lower end of which combustion gases from a sulphur or iron pyrites kiln enter and flow in counter-current. Kerosine, gas oil and lubricating oils absorb about ½ per cent. sulphur dioxide. When a plant is set in operation the quantity of liquid sulphur dioxide required for treating need only be supplied once as all working losses are made good in the manner as described. H. S. G.

Fuel Oil.

303. Process for the Manufacture of Synthetic Liquid Fuels. *Soc. Internationali des Procédés Prudhomme Houdry.* E. P. 275,585. April 4, 1928.

This is a process for the manufacture of liquid fuel, at atmospheric pressure, in which gases and vapours derived from tars, heavy oils or other material,

are led through reaction chambers in which a saturation agent, such as hydrogen, water gas or residual gas is injected after having been previously rendered active by passage through a metallic, oxy metallic or other filter, which has caused it to pass from the molecular state to the atomic state, the reaction between the gases to be saturated and the saturation agent thus being effected without a catalyst.

The apparatus consists of a reaction chamber or chambers, vertically situated, comprising walls perforated or slotted and surrounded by annular cells containing the atomic rendering filters through which the saturation agent is distributed. The atomic rendering material consists of a carrier such as pumice stone on which nickel or an oxide of nickel is deposited.

R. P.

304. Process and Apparatus Permitting the Use of Heavy Oils in Internal Combustion Engines. Haseltine, Lake & Co., for Société Anonyme Le Carbone. British Patent No. 264,407. February 10, 1928.

This patent refers to the use of a heated catalyst at a point in the induction system of an internal combustion engine, so that atomised heavy oil fuel may be broken down to a mixture of vaporised light fuels and atomised heavy oil.

The catalyst referred to consists of activated carbon and is in general formed into a block and maintained at a temperature higher than 250°C. This block may be interposed between the carburettor and the engine, or may be incorporated in the carburettor itself. Passages may be formed in the block through which air and heavy fuel will pass and be subjected to catalytic action on the walls, or the mixture may be constrained to pass through the body of the block itself. Where the catalytic block is incorporated in the carburettor, fuel may be supplied in such a manner that the catalyst is, in parts, saturated with oil. The air supply may then be constrained partly to pass through passages and partly through the material. The block is heated by jacketing and owing to catalytic action light combustible vapours are released. The combined action of air flowing through the passages and air flowing through the block carry to the engine the light vapours and also atomised heavy oil.

The mixture as supplied to the engine is readily ignited by any of the usual methods by reason of the presence of light vapours. E. R. B. S.

Lubricating Oil and Wax.

305. Mercury Heated Vacuum Stills Yield Superior Lubricating Oils. E. N. Klemgard. *The Refiner*, 1928, **7**, 2, 57.

By the process of producing lubricating oils by high vacuum distillation in mercury vapour heated stills no chemicals are used. The reduction in processing cost is at least 25 per cent. The liquid mercury is vaporised in a special boiler so designed that most of the vaporisation takes place in vertical steel tubes closed at the bottom and open at the top and about 4 in. in diameter. The liquid mercury enters the top end of the tubes and drops to the bottom where, when it has reached a temperature of 680-700° F. (depending upon the pressure maintained), it is vaporised and passes out of the heating tubes into a vapour chamber. The system is maintained under a pressure of 8-10 lb. per sq. in. The mercury vapour passes then to a series of 10 reboilers where the oil is heated to its boiling point. The mercury vapour passes in counterflow to the oil being fractionated. Thus

the vapour first enters the reboiler in which the heaviest oil is being fractionated. As the mercury gives up its heat to the oil a portion of it is condensed and drops to the bottom of the mercury chamber of the evaporator, where it enters a pipe line and flows by gravity back to the mercury boiler. The mercury vapour which is not condensed passes up to the next evaporator and so a portion passes through the series of 10 evaporators in the last of which hot, caustic pre-treated topped crude is being pumped.

Topped Smackover crude is heated with from 1-2 per cent. of a solution containing 50 per cent. NaOH in a shell still. This crude, before being treated with the caustic, has an organic acidity equivalent to 0·8 mg. KOH per g. of oil. It is believed that the crude contains about ½ per cent. of naphthenic acids, and members of the series having boiling points of 380-650° F. no doubt exist in the crude. One of the reactions taking place between the caustic soda and the oils is known to be the neutralisation of the naphthenic acids. However considerably more caustic soda than is required for the neutralisation of these acids is used and during the distillation reacts with some of the hydrocarbons. This is indicated by the production of a considerable proportion of relatively unsaturated low-boiling oils having a very foul odour and termed "stink oils." The sodium compounds of these unidentified hydrocarbons are apparently decomposed during the distillation and in some cases a deposit of caustic soda and sodium is left in the evaporators and still. In one instance sufficient sodium was formed to react with water to form hydrogen when the still was being cleaned.

The oil, after being vaporised by the mercury in the evaporator sections, passes upwards into fractionating columns, one fractionating section being provided for each evaporator. In some cases two or more of the fractionating sections are joined at their upper ends by horizontal sections which may carry refluxes. Projecting upwards from the horizontal sections are additional fractionating units which may be termed stink-oil separators. From the top of each are pipe lines through which the stink-oils pass to heat exchangers and condensers. From near the bottom of the stink-oil separators the finished oils run to receivers. The entire system is maintained under a pressure of 6·7 mm. of mercury. S. W. C.

306. Method of Decolorising Oils under Pressure. P. W. Prutzman, Assr. to Contact Filtration Co. U.S.P. 1,653,735, Dec. 27, 1927.

The invention provides for a continuous method of purifying and decolorising oils with adsorbents which comprises continuously heating a confined flow-stream composed solely of an oil to be decolorised and an adsorbent to a temperature above the vaporisation point of the oil while applying pressure to the stream to prevent the evolution of oil vapour, cooling the stream below the vaporisation point of the oil, and reducing the pressure on the stream after cooling. Thereafter the adsorbent is mechanically separated from the cooled oil. S. W. C.

307. Wax Sweating and Crystallising Apparatus. H. L. Allan and J. Moore, Assrs. to Burmah Oil Co., Ltd. U.S.P. 1,654,232, Dec. 27. 1927.

The invention comprises an apparatus for crystallising and sweating paraffin and like wax comprising a plurality of superimposed and spaced heating or cooling cells, a casing enclosing the cells, a cylindrical ring depending from each cell to provide intermediate wax compartments, a raised perforated wax support in each compartment, each ring resting upon the next lower perforated wax support, a vertical wax and oil supply and discharging chamber alongside of and outside the periphery of the cells which opens

to the interior of the compartments, each ring having an opening adjacent to the charging chamber and an air vent diametrically opposite the opening and close to the underside of the next higher cell. S. W. C.

308. Process of Reconditioning Crank Case Oil. L. L. Odom, Assr. to Purity Chemical Co. U.S.P. 1,655,660, Jan. 10, 1928.

The invention provides for a process of reconditioning used crank-case lubricating oil containing diluents. The low boiling diluents are distilled off, the residual oil cooled and filtered, further cooled and agitated at a temperature below 95° C. with sulphuric acid for about 30 min., washed with an equal volume of water, heated and agitated at a temperature of approximately 200° F. with a solution of NaOH for about 30 min., washed with an equal volume of water and heated to evaporate off the intermingled water, subsequently agitating with diatomaceous earth and filtering, whereby purified lubricating oil is produced. S. W. C.

310. Process of Refining Oil. J. C. Black. U.S.P. 1,656,997, Jan. 24, 1928.

The invention provides for a process of refining hydrocarbon oil comprising commingling an acid treated pulverised adsorbent clay and oil, subjecting the mixture to agitation and heat, regulating the degree of heat for that particular oil whereby a rapid reaction of the clay with the oil results, then partially cooling the mixture to a point below rapid oxidation, then separating the oil and clay and finally cooling the oil. S. W. C.

311. Wax Sweating and Crystallising Apparatus. Burmah Oil Co. (from Hugh Logie Allan and James Moore). E.P. 287,714. Mar. 26, 1927.

This relates to improvements in British Patents Nos. 208,195 and 243,447. An upright vessel for the heat-treatment of wax is divided into superimposed wax compartments by transverse dished or conical partitions, each compartment being provided with a series of transverse pipe coils, a perforated wax support below the coils, a central pipe for charging air-free wax and means for the escape of the air displaced during charging. The pipe coils may be of uniform radial pitch throughout; evenly spaced vertically; or with a radial pitch of 2 to 4 inches and spaced at vertical intervals of 2 to 4 inches. Means may be provided for supplying wax mixture to fill the compartments during the cooling phase. The pipe coils may have their lower coils more closely spaced than the upper coils. The transverse partitions may consist of a solid dished plate through the centre of which the charging pipe opens so that wax cannot descend to the next lower compartment except by the pipe, which also serves as a discharge pipe. Steam or water jets may be provided around the space beneath each wax support, and a heating pipe may also be provided. The shell of the vessel may be so constructed as to form the side of the conical trays and form enclosed trays open only to the central pipe. The wax mixture may be fed by means of a vertical header provided with branch pipes at the top of each compartment and a steam pipe may be inserted in the header. The coils of each series may be staggered in relation to each other and afford more support for the wax cake. The jets supplying steam to the space below each wax support also enable the space to be washed out. G. S.

312. Manufacture of Lubricating Oils. F. W. Hall, Assr. to Texas Co., New York. U.S.P. 1,668,313. May 1, 1928.

In the manufacture of lubricating oils, difficulty is experienced in treating cylinder stocks with sulphuric acid owing to incomplete separation of the acid

sludge products of the reaction and on account of the refractory emulsions which are formed in the ordinary neutralisation processes. Efforts have, therefore, been made to refine cylinder stocks by first diluting the oil with a light petroleum distillate and then subjecting the diluted oil to the action of acid. This method, however, results in heavy loss of acid due to reaction with the diluent as well as the cylinder stock, and difficulty in handling the soft resultant sludge. By the present process cylinder stock can be satisfactorily treated by reaction of the undiluted oil with acid and then adding a diluent. When the viscous oil is treated with acid a hard and tenacious sludge is formed not readily soluble in cylinder stock and light oil, so that when the light oil is added, after the acid treatment, the sludge products readily settle out. After the separation of this sludge the oil may be neutralised with concentrated alkaline solution without the formation of tenacious emulsions. By this means cylinder oils of low carbon residue may be obtained with minimum quantities of acid.

As a typical instance of the above treatment 1,000 barrels of 600 fire stock is treated with 40 lb. per barrel of 98 per cent. sulphuric acid. An equal volume of naphtha is subsequently added, the mixture blown with air and the sludge allowed to settle. The oil is removed to another vessel and treated with about three barrels of concentrated caustic soda solution. After settling the oil is agitated with a small quantity of fine fuller's earth. The earth is removed and the oil distilled with fire and steam to 600° F. fire test.

H. S. G.

313. Decolorizing Oils at High Temperatures. P. W. Prutzman, Assr. to Contact Filtration Co., California. U.S.P. 1,667,984. May 1, 1928.

A method by which oils may be treated with absorbents at temperatures above the vaporization and boiling point without loss of vapour. The apparatus consists of a tank in which oil and absorbent are mechanically mixed and from which oil is withdrawn by a pump and forced into a heater coil set in a combustion chamber. The oil passes on to a cooler comprising a water-jacketed pipe in which the temperature is reduced sufficiently to condense whatever vapour is generated in the heater by cracking or otherwise. From this cooler the oil passes into one of two branch pipes controlled by valves and leading to steamers. As one steamer is being filled steam is diverted to the other. Each steamer is filled about two-thirds full with hot oil. Steam is admitted into the body of the oil and withdraws vapours of volatile cracked products and carries them into a vapour line leading to a condenser. When steaming is complete, a valve in the bottom of the steamer is opened and the contents pumped to a filter press. Whilst the first steamer is being emptied the second comes into operation, and the process thus becomes continuous. Any class of oil may be successfully treated by the above method, even in crude condition or acid treated and washed. If it has been acid treated and left in an acid condition the absorbent will effect a complete neutralization of the oil coincident with decolorization and purification.

H. S. G.

314. Process for the Preservation of Mineral Oils. F. Hofmann. E.P. 289,909. 1928.

The darkening and sludging of oils, especially lubricating and transformer oils, by oxidation, can be largely inhibited by adding to the oil a cyclic amine of high alkalinity. Piperidine, diethylaniline and phenylene diamine are specifically mentioned.

H. G. S.

315. Process for Distilling Oils. Harnsberger. U.S.P. 1,666,597. April 17, 1928.

The process refers to the distillation of petroleum for the production of lubricating oils.

Oil is pumped into a preheater coil of a conventional pipe still and passes into a separating chamber where distillates too light for lubricating stocks vapourize and the preheated stock is passed back for redistillation, in a pipe still of novel design. This consists of a bank of tubes of increasing area, the increase required being calculated from the amount of vaporization caused by the heating under the chosen vacuum. It is thus claimed that undue back pressures due to vaporization and consequently local overheating and cracking are avoided. The oil now passes to a series of separating towers fitted with dephlegmators where it is fractionated as desired.

Very heavy distillates of good colour and flash point are claimed to be produced at a vacuum of 12 in. of mercury on the outlet of the still, and the plant is inexpensive in primary cost and operation. A. R. S.

316. Manufacture and Treatment of Lubricating Grease for Use with Grease Guns. C. Brower. E.P. 288,796. March 17, 1927.

The object of the invention is to provide a good lubricating grease in a convenient form and one which will retain an almost solid consistency between very wide differences in temperature, as for example —18° F. and 340° F., allowing handling in block or bar form without any covering.

In the manufacture, the steam distilled lubricating distillate of a suitable paraffin base crude petroleum oil, is combined at a temperature of 300° F. with a suitable stearine or stearine compound in the proportions of 9 : 1. The combined liquid is distilled in a cast iron still with a long cast iron worm condenser by means of superheated steam in a current of oxygen for 12 hours. The residue is immersed in benzine and insoluble asphalt allowed to deposit, and removed. The remaining solution is distilled, leaving a semi-transparent grease-like mass which is redissolved in the condensed product of the first distillation. On cooling the mass solidifies to a firm semi-transparent grease of exceptional "oiliness" and of a melting-point about 350° F.

The grease may be moulded into the shape of a grease gun refill or "candle" and provided with a longitudinal groove of V shape. For convenience in handling; the candle may be coated with a layer of high melting point paraffin wax, and also cut to any convenient size. With a grease gun charged with one of the above candles, the lubricant ejected from the nozzle has the consistency of a soft grease such as petroleum jelly, owing to the pressure exerted in using the gun. By virtue of the absence of air locks it is easy to fill the gun completely with lubricant. H. S. G.

Special Products.

317. Cellosolve and its Derivatives in Nitrocellulose Lacquers. E. W. Reid and H. E. Hofmann. *Ind. Eng. Chem.*, 1928, **20**, 497–504.

The ethers of glycol, notably the mono-ethyl ether "cellosolve," have found wide use in the lacquer industry on account of their high solvent action on nitrocellulose, their high dilution ratio with hydrocarbons and their low price. Other derivatives, such as cellosolve acetate (monomethyl ethyl acetate) and Butyl cellosolve (the corresponding butyl ether), are also good nitrocellulose and resin solvents. All have a slow rate of evaporation owing to their high boiling points. This latter property is a distinct advantage, as the last portion of the solvent mixture to evaporate is a good solvent for non-volatile ingredients and therefore gives a clear homogeneous film. All three compo unds have little or no odour and are particularly useful for the finishing

coat of articles where a disagreeable residual odour is a disadvantage—*i.e.*, cooking furniture. An important property of these solvents is the fact that, even mixed with comparatively high percentages of aromatic hydrocarbons, they are still capable of acting as nitro-cellulose solvents. The chief advantage of cellosolve acetate, however, is its ability to prevent "blushing" under humid conditions, and it is owing to this property that it finds its greatest use in high-grade automobile lacquers. Butyl cellosolve, on account of its high boiling point, is particularly suitable for brushing lacquers and, owing to its slow solvent action, for lacquers to be used on old varnish and paint. The boiling points of cellosolve, cellosolve acetate and butyl cellosolve are 134°, 153°, and 170° C. respectively. S. F. B.

318. Improved Method of Recovering Acid and Resinous Matter from the Acid Sludges Formed in the Refining of Mineral Oils. W. Demann. E.P. 258,241. Jan. 25, 1928.

The invention provides for a method of purifying untreated acid sludge from the refining of benzole consisting in stirring the acid sludge, cooling if required, with about 30 per cent. of water and 30–50 per cent. of heavy benzine or tetrahydronaphthalene or like organic substances of a strongly aliphatic or polymethylene character and centrifuging to obtain resin almost free of acid and solvent or drawing off the fluid and then centrifuging the resin, finally grinding the separated resin with gas liquor, ammonia, caustic soda or other alkali. The separated resin if solid is dried and ground, and if liquid is stirred with alcohol or other carriers or turpentine oil or other driers to yield a slowly drying hard lacquer. S. W. C.

319. Making Bitumen-pitch Type Emulsions. L. Kirschbraun. E.P. 286,844. Jan. 31, 1927.

A process of making aqueous emulsions of the bitumen-pitch type in which a thick pasty or plastic mass is first secured by partly filling an emulsifier with water, starting the mixer shaft and adding an emulsifying agent, such as clay or bentonite, gradually until the mixture is like mud and is undergoing viscous circulation, preferably downwardly through an inner open-ended cylinder mounted in the emulsifier and upwardly in the annular space between the cylinder and the wall of the emulsifier. The aqueous suspension of emulsifier thus prepared is brought to a temperature approximating the melting-point of the bitumen-pitch type base to be used, which is heated to a temperature ranging from 200° to 450° F., and added to the plastic suspension in the emulsifier. Under the action of the agitator, dispersion takes place and agitation is continued until the proper proportions of water, emulsifying agent and base, and the desired consistency of the emulsion are established. The temperature is maintained the whole time at approximately the melting-point of the base used. After production is established in the emulsifier, regulated and controlled quantities of base and aqueous suspensions of emulsifying agent are introduced and controlled quantities of end product withdrawn, the amount withdrawn being equal to the volume of ingredients introduced. H. S. G.

320. Bituminous Emulsions. J. A. Montgomerie. E.P. 288,821. April 23, 1927.

A development of E.P. 226,032 for the production of aqueous bituminous emulsions by mixing directly melted asphalt and a hot aqueous caustic alkaline solution, in which a bicarbonate or carbonate is substituted for the caustic alkali. As an example, an emulsion is prepared from 600 parts by weight of Mexican asphalt, 4 parts of potassium carbonate, and 400 parts of water in which the carbonate is dissolved, by pouring the molten asphalt into the hot dilute potassium carbonate solution. A small percentage of

soap may be added to the resultant emulsion to give increased spreading or wetting power. H. S. G.

321. Manufacture of Asphaltic Bituminous or Tarry Emulsions. Société Anonyme "La Trinidad." E.P. 264, 496. Jan. 11, 1927. Conv., Jan. 12, 1926.

A process for the manufacture of asphalt, bitumen or tar emulsions, intended for the construction and maintenance of roads and pavements, consisting in first preparing a mastic by mixing with a natural or artificial asphalt, bitumen or tar binder, neutral pulverulent materials such as cement or ground limestone, then emulsifying the mastic by mixing it with a suitable aqueous solution of an emulsifying agent such as soap. H. S. G.

Analyses and Testing.

322. A Variation of the Carius Method for the Determination of Sulphur. *J.A.C.S.*, 50, IV, 1231.

Carius has suggested the addition of a halogen body to the fuming nitric acid due to the fact that halogenated bodies are more easily oxidised. Kupp introduced a barium salt and found a marked shortening of the time to gather the resulting sulphate. The authors, however, do not recommend this latter, due to the liability of obtaining finely divided glass resulting from the breaking of the tube in the precipitate. Results of analysis are given using 0·1 gm. sulphonal and 3 ccs. fuming nitric acid heated at 200° C. to allow a large time of oxidation. In series II four drops of bromine was used, and in series V a 5 per cent. excess of barium chloride, and in III, IV and VI an excess of salt corresponding to series V.

Percentage of Theoretical Sulphur appearing as Sulphate.

Time of heating. hours.	I. HNO_3	II. HNO_3+ Br_2	III. HNO_3+ $BaBr_2$	IV. HNO_3+ $Ba(No_3)_2$	V. HNO_3+ $BaCl_2$	VI. HNO_3+ NaCl.
6	14·2	6·6	22·8	20·1	50·7	32·1
12	25·4	23·4	41·1	44·0	75·6	75·6
24	46·2	49·6	57·8	66·4	84·4	95·6
48	70·6	71·7	72·1	85·6	95·2	99·5

For the oxidation of stable sulphones the use of 0·3–0·4 gms. of sodium or potassium chloride with 3 ccs. of fuming nitric acid is suggested. After heating, cooling and opening, the contents of the tube with the washings can be filtered and evaporated to dryness, provided sufficient alkali salt has been added to fix the sulphate. The analysis is then finished in the usual manner. A. R. S.

323. Burette Reading Device. Hyman. *Chem. and Ind.*, 47, XIV, 100 T.

The general method of reading a burette by holding a white card behind the level affords no assistance in avoiding parallax. To rectify this the author uses a piece of transparent celluloid 3 ins. × 1½ ins., the lower two-thirds of which are blackened. Another piece of transparent celluloid is fixed by the ends to this so that the upper edge of the latter coincides with the upper blackened edge of the former. The device is then prised open and slipped over the burette. The slightest movement of the eye throws the edges of the device out of alignment, while very slight lowering or raising of the device causes the bottom of the meniscus to appear above or below the black line. Of nine independent observers a reading of 6·03 was given by seven and of 6·02 by two on test. A. R. S.

324. LIQUID-STANDARDS FOR VISCOMETERS. D. Krüger, *Zeit. für Angew. Chem.* **41**, **15**, 375.

The choice of a liquid for standardisation purposes of technical viscometers depends upon the type of viscometer and the nature of the liquids with which it is to be used. Whilst water is used almost exclusively in the case of narrow capillary viscometers such as those of Ostwald and Hess, which are used for the investigation of volatile fluids, still more volatile liquids which can be obtained sufficiently pure—*e.g.*, acetone and ether—are also suitable.

For the more common viscometers, which are used for such viscous liquids as oil, gums and glues, water is unsuitable for standardisation purposes. For these viscometers castor or other oils are more generally used as standards. Considerable variation in the viscosity of different samples of these standard oils exists and depends upon the method of preparation used and subsequent changes in the oils under the influence of light and air. Variations to the extent of 2–3 per cent. occur in the values given in the literature for castor oil.

Absolute Viscosity of Castor Oil (Centipoises).

	Hyde.	Kahlbaum and Raber.	Gilson and Jacobs.
20° ..	10·15	9·86	9·9
30° ..	4·68	4·51	—

Concentrated glycerin solutions, being highly hygroscopic, are unsuitable, but solutions of cane sugar appear to be more appropriate. Solutions of the latter up to 40 per cent. have been examined by various authors, and Bingham and Jackson have examined solutions up to concentrations of 60 per cent. and for temperatures from 0–100°. Some data are appended.

Absolute Viscosity of Cane Sugar Solutions (Centipoises).

Temp.	Per cent. by weight of cane sugar.					Observer.
°C.	10	20	30	40	60	
15	—	2·267	—	7·468	74·6	
20	—	1·960	—	6·200	56·5	Bingham and
25	—	1·704	—	5·187	43·86	Jackson.
15	—	—	—	—	67·6	Calculated from Mühlendahl's formula.
20	—	—	—	—	49·8	
25	—	—	—	—	36·75	
15	1·518	2·212	—	7·30	—	
20	1·328	1·910	—	6·07	—	Hosking.
25	1·173	1·674	—	5·08	—	
20	1·33	1·89	3·24	—	—	Powell.
25	1·17	1·70	2·75	5·12	—	
20	1·362	1·934	3·140	—	—	Burckhard.
25	—	1·731	—	—	—	Bingham, Schlesinger and Coleman.

The data of Bingham and Jackson are probably more reliable than those of Hosking. In measurements of viscosity the use of the same standard of known definite viscosity is necessary. T. A. S.

325. DETERMINING SULPHUR IN HEAVIER OILS. Devine and Lane. *Oil and Gas Journal.* May 1, 1928, 164.

The oxygen bomb method of determining sulphur in oils has been said to give lower results than should have been obtained.

The authors therefore analysed heavy oils containing 2·5 per cent. to 0·15 per cent. of sulphur by both bomb and Carius methods, when it was

found that the variation calculated on the total sulphur present amounted to 2·47 per cent. for the highest sulphur content and increased to 7·3 per cent. for the oil containing 0·15 per cent.

Medicinal oil was treated with flowers of sulphur until dissolved, and this was used in varying dilutions of medicinal oil for analysis by both the bomb and Carius methods, with the following results.

Per Cent. of Sulphur Found.

Sample	1	2	3	4	5
Carius method	2·55	1·12	0·624	0·299	0·150
Average of six bomb determinations	2·47	1·08	0·598	0·282	0·139
Per cent. difference	3·1	3·6	4·2	5·7	7·3

The addition of ammonium nitrate has been suggested to aid the oxidation of SO_2 to SO_3 during combustion in the bomb and thereby increase the percentage of sulphur found. The authors find that this addition does not materially increase the amount of sulphur as determined. A. R. S.

326. Rapid Corrosion Test for Gasoline. Rue. *Oil and Gas Journal.* May 10, 1928, 162.

In the sweetening of gasoline by the plumbite method, the addition of the excess of free sulphur with the plumbite used is dissolved in the spirit, causing the final product to be corrosive. It is, therefore, obvious that if proper control of the process is to be kept, a rapid and sensitive control test must be frequently made.

Up to the present time this control has been made by immersing a polished copper strip at temperatures about 200°F. for two to three hours, the exact time and temperature varying in different refineries. The authors take exception to the time required for this test and prefer to shake 100 ccs. of treated gasoline with 1 cc. of clean metallic mercury for 5 minutes. The liquid is then decanted through a qualitative filter paper which is dried and examined for sulphide precipitate.

The table shows this method to be more sensitive than the copper strip method.

Copper Strip Test.

Copper Sample	Strip method lbs. free sulphur 1,000 bbls.	3 hrs. at 200°F.	½ hr. at 200°F.	3 hrs. at 122°F.	Mercury test	Doctor test
1	0	Slight	Negative	Negative	Slight	Positive
2	5	do.	do.	do.	do.	do.
3	10	do.	do.	do.	do.	do.
4	15	do.	do.	do.	do.	do.
5	20	do.	do.	do.	do.	do.
6	25	Negative just noticeable	do.	do.	do.	Negative
7	30	do.	do.	do.	This and	do.
8	35	do.	do.	do.	following	do.
9	40	do.	Slight trace	do.	showed	do.
10	45	Slight	do.	do.	very black	do.
11	50	do.	do.	do.	precipitate	do.
12	55	do.	do.	do.		do.

A. R. S.

327. THE RECOGNITION OF ADDED PARAFFIN IN CERESINE. Holde and Schünemann. *Zeit. für Angew. Chem.* 41, **15**, 368–375.

Ceresine, obtained by treating crude ozokerite with sulphuric acid, is frequently adulterated with paraffin. Since ceresine consists largely of iso-paraffins the presence of added normal paraffin can be detected. More exact refractometric methods have replaced the old tests of kneading and touch.

Ulzer and Sommer determined the refractivity of paraffin in terms of the Zeiss butter-refractometer scale at 90°, showing that with rise of melting-point (42–68·8°) the value rose from −7·2 to +6·5. This value is termed n_{90}. In the case of pure ceresine there was a corresponding rise in the value of n_{90} with rise of melting-point from +7·6 to +19·2. The wide range of these values has led to the development of a combined fractional precipitation and refractometric method which enables the presence of 10 per cent. of paraffin of m.p. 53° to be detected.

Ceresine (2 grm.) is dissolved in 60 ccm. chloroform in a wide-necked erlenmeyer flask (300 ccm.), fitted with a condenser. Alcohol (120 ccm. of 96 per cent) is now added, the mixture being well stirred with a glass rod and the closed flask kept at 20° for 10–15 mins. The precipitate is collected on a porcelain filter, the portion adhering to the walls of the flask and the glass rod being removed by means of a soft brush without the addition of further solvent. The precipitate is pressed free of solvent and then washed with hot benzol into a weighed dish. This is called precipitate No. 1. The mother liquor from this precipitation is evaporated, re-dissolved in 10 ccm. chloroform, and again precipitated by the addition of 30 ccm. 96 per cent. alcohol. This precipitate (No. 2) is treated as above. The filtrate, after removal of the solvent at 105°, gives residue r, which is weighed and its refractive index taken. It is then re-dissolved in 5 ccm. chloroform and pre-cipitated with 20 ccm. of 96 per cent. alcohol giving precipitate 3 and residue r^1. The refractive indices of the precipitates and residues are determined at 100° and corrected to terms of the butter-refractometer scale at 90°. The presence of paraffin becomes apparent in precipitates 2 and 3 but is more clearly shown by the relation $(n_3 + nr^1)/gr$ where gr is the percentage by weight of the residue r. This value, denoted in the table by Q, is in the case of added paraffin less than 3·3, the lowest value for normal ceresine.

TABLE I.

Fractional Precipitation of Pure Ceresine and Paraffin-Ceresine Mixtures.

	Precipitate No. 1.		Precipitate No. 2.		Residue r.		Precipitate No. 3.		Residue r^1.		Q.
	%	n_{90}	%	n_{90}	%	n_{90}	%	n_{90}	%	n_{90}	
Pure ceresine ..	76·7	8·7	1·5	3·8	3·3	13·6	0·4	2·7	2·1	17·2	3·3
	to	to	to	to	to	to	to	to	to	to	to
	93	17·9	11·4	14·1	8·4	31·2	1·3	8·9	7	33·9	8·1
Ceresine + 10%	71·5	8·6	5·6	2	5·3	4·5	0·8	0·1	3·6	6·2	1·3
paraffin m.p.	to	to	to	to	to	to	to	to	to	to	to
53°	84·5	17·5	14·4	5	9·7	16·8	2·1	3·3	8·2	20·3	2·6
Ceresine + 20%	75·5	12·4	9·7	2	11·5	11·5	2·1	−0·1	8·7	15·4	1·35

The characteristics of various ceresines are given in the Table II.

TABLE II.

A.—*Guaranteed Ceresines.*

Source.	M.-p. °C.	n_{80}.	Colour.	Residue r. %	Residue r. n_{80}	Precipitate No. 3. %	Precipitate No. 3. n_{80}	Residue r^1. %	Residue r^1. n_{80}	Q.
Polish ..	60—62	14·5	Lt. yellow	6·3	26·1	1·6	11·4	4·6	33·2	7·1
„ ..	60—62·2	13·5	White ..	6·7	23·2	1·7	10·4	4·9	30·1	6·0
„ ..	74	14·8	Yellow ..	7·2	15·8	1·9	6·4	5·3	19·6	3·6
Caucasian	75—77	21·6	White ..	19·8	34·2	4·3	35·0	15·5	33·9	3·5
Polish ..	77—78·8	18·2	White ..	15·0	27·3	3·7	20·1	11·3	31·4	3·4
„ ..	82—84	24·3	Yellowish brown	7·2	40·3	2·8	17·8	4·4	55·0	10·1
„ ..	83—86	20·9	Lt. yellow	4·5	20·4	2·1	13·8	2·3	44·4	13·0
„ ..	85—87	23	Ochre yellow	4·6	24·1	1·5	11·1	3·1	31·8	9·3
B.—*Unsuspected Ceresines.*										
Polish ..	60—61	13	Pale yellow	5·0	18·5	1·6	8·0	3·4	22·9	6·2
„ ..	70—71	20·3	White ..	7·0	20·0	1·7	26·0	5·4	19·0	6·4
„ ..	71—72	24	Yellow ..	6·3	36·3	1·6	35·0	4·6	38·0	11·7
„ ..	72·5—74	16·8	White ..	13·7	24·5	3·1	17·1	9·9	27·9	3·3
Rumanian	75—76	22·1	Lt. yellow	4·6	46·7	0·9	43·3	3·7	47·7	19·7

From the benzene insoluble hard asphalt of an American cylinder oil, a ceresine has recently been extracted having the high melting-point of 81–82·5° and $n_{80}=25·6$.

The characteristics of the waxes used as ceresine adulterants are given in Table IV.

TABLE IV.

Type of wax.	M.-p. °C.	n_{80}	Residue r. %	Residue r. n_{80}	Precipitate 3. %	Precipitate 3. n_{80}	Residue r^1. %	Residue r^1. n_{80}	Q.
American table ..	50—52	2·85	16·1	1·7	8·1	1·5	8·0	1·9	0·21
American scale III. ..	50—52	3·0	16·4	2·0	7·3	1·4	9·1	3·7	0·31
Rangoon table ..	51—53	3·0	12·9	1·2	4·7	0·4	8·2	1·8	0·17
German	52—54	1·5	20·6	1·0	7·0	0·4	13·6	1·9	0·12
American scale IV. ..	52—54	2·0	12·2	4·1	4·0	0·4	8·2	6·2	0·54
„ II. ..	52—54	3·0	16·5	3·2	6·1	0·1	10·4	4·8	0·3
„ I. ..	52—55	4·0	14·4	5·7	4·8	2·1	9·6	8·2	0·71
German	53—55	1·3	10·8	0·7	3·4	0·6	7·4	2·0	0·24
Polish table	54—56	4·25	11·8	1·5	4·9	1·4	6·9	1·6	0·25
Rangoon table ..	58—60	5·2	7·0	2·1	2·1	2·8	4·9	3·1	0·84
„ ..	74·5—75·5	13·6	3·4	28·7	0·45	15·4	3·0	31·8	13·9

These waxes, with the exception of the abnormally high melting Rangoon paraffin, all give lower values for Q (*e.g.*, 0·1–0·8) than is obtained with ceresine. The viscosities of ceresines, determined by the Vogel-Ossag viscometer, vary from 8·3 to 10·7, whilst those of paraffin waxes lie between 4·6 and 5·5 except in the case of high-melting Rangoon wax, which is 9·7. The dispersion co-efficients for ordinary waxes lie between 63·9 and 64·1, whilst those for ceresines are lower, lying between 63·85 and 63·68. There is considerable loss when ceresines are treated with chlorsulphonic acid, the isoparaffins being attacked. The loss with paraffin is considerably less. Similar action occurs when ceresines and waxes are treated with fuming sulphuric acid. Tables are given showing the changes in the constants which result from this action. Tables are also given showing the constants of ceresine

and paraffin mixtures when examined by the method of fractional precipitation. The weight of the fractional precipitates 2 and 3 increases with increase in the quantity of paraffin. This is particularly marked in the case of fraction No. 2. The constant $Q^1 = \frac{n_{20}F_2 + n_{20}F_3}{gF_2 + gF_3}$ falls within the following limits:—

Percentage added paraffin	=	0	5	10	20	30	40	50
Q^1 ..	=	1·9—3·2	1·2—1·7	1·2—1·7	0·6—1·1	0·5—0·9	0·4—0·9	0·2—0·7

T. A. S.

Refinery Plant.

328. TEMPERATURE MEASUREMENTS IN OIL TANKS. By H. Allen Hamburg. *Petr. Zeit.* Vol. 24. No. 11, p. 481.

The temperature coefficient of oils is very high hence the temperature is not homogeneous in tanks containing oil. The determination of "mean tank temperature," *i.e.*, the average temperature which the oil assumes, if all temperature differences are equalised, is sought in this article.

Regarding the *theories of "mean tank temperature,"* a cylindrical tank is filled with oil to a definite height H which is heated by heating coils 1 metre from the bottom of the tank. A rod L dips into the oil-column and the distribution of temperature is represented along this rod (as shown in fig.). The height of the oil coil h is represented on the abscissæ and the corresponding increase of temperature t by the ordinates; dh. is interpreted as an infinitely small increment in the height of the tank represented geometrically by means of a perpendicular on the temperature curve. To every such portion of the surface a definite temperature t is ascribed, its content being t, dh. The sum of all such perpendiculars between the bottom of the tank O and the oil surface H gives the surface below the temperature distribution curve

$$F = \int_0^H t.\,dh.$$

Dividing this surface by H we have the mean tank temperature

$$\int_0^H t.\,dh/H.$$

Thus the latter can be calculated assuming the temperature distribution curve but this curve is only an instantaneous photograph. The temperature distribution assumes another value at another moment even if the average value remains the same, and in the calculation of the tank content only that tank temperature is determinative which has prevailed at the moment of measurement of the oil level. The "mean tank temperature" is accordingly defined as the mean height of the temperature distribution curve at the moment of measurement.

Practical Determination of Temperature Measurement. (a) *Differential Process.*

This consists of withdrawing as many as possible height intervals of oil by means of a test can and measuring the temperature of same. Theoretically it would then be possible, assuming the individual samples to be infinitely small, to obtain a temperature distribution curve and so derive the mean tank temperature from it. There are, however, several fallacies in this process.

The differences are finite and very large compared with dh, and the mean tank temperature can only be calculated from the sum of the measured temperatures divided by the number of measurements. A further deviation is the time taken to carry out the observations and the failure to acquire an instantaneous diagram of the temperature distribution. There is also the danger that the measured temperature of the sample may not be identical with that which prevailed in the spot in question in the tank. The heat lost (Q) during the withdrawal of the sample from the tank and the observation of the thermometer, also radiation losses.

Q is expressed by $K.Z.F._s(t_{oil} - t_{air})$ where K= heat coefficient in K cals./m. hr. and 1° C. To make Q as small as possible each of these 4 factors must be made as small as possible.

The *heat coefficient K* is rendered small by insulating the test can with asbestos and spreading over with a suitable water-tight cement (water glass, clay cement), thus presenting a smooth outer surface.

The *time t* is made slight by removing the sample quickly and reading its temperature immediately.

The *surface F* depends on the content of the vessel which usually amounts to ¼—½ litres. The height of the vessel compared with the depth of immersion of the thermometer should not exceed 20 cms. The best surface is cylindrical with an orifice of very small cross-section.

The *temperature difference* (t_{oil}—t_{air}) is determined by external conditions. If the difference is nil, Q is nil, but this case is rare. The difference is considerable in frosty weather, then the temperature of the sample must be measured in a protected spot and the test vessel must be held in the manhole. With liquids with a high vapour tension like benzene, benzol, spirit, etc. t_{air} can sink considerably below the surrounding temperature by cold due to evaporation on the surface of the cans. In such cases the liquid adhering to the test can can be removed immediately after withdrawal, by drying.

(b) *Maximum-Minimum Thermometer.*

This thermometer is either applied for a length of time in the tank or drawn slowly through the oil-columns from below upwards. The maximum should then indicate the highest, the minimum the lowest temperature of the tank content, and the mean of the two values gives the mean tank temperature.

Theoretically the numerical values of the temperature differences occurring in the oil are not determinative, but only the *distribution of temperature*. The maximum thermometer gives two temperatures—the highest and the lowest—but does not specify where these occur. As a result of this no conception can be formed of the temperature distribution curve, and there is no foundation for the calculation of mean tank temperature. However the effect on a cold day, or with excessive cold by evaporation is considerable with the minimum reading due to temperature differences.

Thus in the performance of a surface tank control it is best to remove a sample from the centre of the tank content and to determine its temperature rapidly and in the manner described. W. S. C.

329. Design of Fractionating Columns. D. B. Keyes, R. Soukup and W. A. Nichols. *Ind. Eng. Chem.* 1928, **20**, 464—6.

The most practicable method of estimating the theoretical number of plates required to separate a binary liquid mixture into its components is that devised by McCabe and Thiele (*Ind. Eng. Chem.* 1925, **17**, 605). This method requires as data the composition in mol per cent. of the feed, the product and the residue and also the reflux ratio, and equilibrium is necessary between vapour and liquid on each plate. To determine the actual number

of plates required, a safety factor known as the plate efficiency is applied to the theoretical number. This safety factor varies greatly with changing conditions and cannot be determined in advance. The authors, however, have devised a simpler graphical method which applies a safety factor, found by experience, to the McCabe and Thiele method and obtain results agreeing substantially with those of other investigators. Thus 20 plates, 45 to 60 plates and 61 to 82 plates are respectively required for the fractionation of the systems benzene-toluene, ethyl alcohol—water and water-acetic acid. H. G. S.

330. One Fractionating Column Handles Vapours from Six Stills. G. Reid. *The Refiner*. 1928, 7, 2, 69.

The six shell stills are operated in continuous series in such a manner that all the vapours coming from the first five stills are delivered into the tower at about its middle through a common vapour line. This vapour line is telescoped, being part 6-inch then enlarged to 8-inch, and then to 12-inch at the point where it begins to carry the largest volume of vapours and enters the tower at the latter dimension. The crude flows through the stills from 1 to 5, each still being carried at a successively higher temperature. The residue leaving No. 5 still has been stripped of everything down to the gas oil fraction. The vapours enter the tower and flow upward, passing counter-current to the condensate of gas oil derived from oil previously vaporised in the stills. Vapours leaving the top of the tower are conducted to Sterling condensers and condensed as gasoline having an F.B.P. around 437° F. The reflux is collected at the bottom of the section and conducted away through cooling coils to storage. This material has an I.B.P. higher than the F.B.P. of the overhead gasoline.

The lower section of the tower, also provided with bubble trays may be termed a "re-boiler." Residuum from No. 5 still is jetted into the tower at about its middle and a little below the bottom separating plate which divides the tower into two sections. This residuum flows downward over a series of bubble plates, leaving the tower at the bottom and returning into the sixth still. Oil in this still is fired at sufficient temperature to vaporise the remaining portion of the gas oil fraction. Vapours leaving this still are conducted to the bottom of the lower section of the column, where they pass upward, counter-current to the residuum jetted into the tower from No. 5 still. This scrubbing action assists in stripping the oil of any light portions remaining in it. Vapours from all stills combine in the lower section of the tower and are conducted through an internal vapour line upward and into the upper section of the column. Thus all the vapours from the entire volume of crude charged are finally fractionated into the two desired cuts of gas oil and gasoline. The residuum is drawn off from the bottom of No. 6 still, cooled by exchange with incoming crude and transferred to storage as fuel oil. S. W. C.

331. Pipe Stills with Pennsylvanian Crude. Alfred Mulhaupt. *Oil and Gas Journal*, 1928. **26** (49), 86, 152.

The size of a pipe still with a single fractionating tower appears to be unlimited and the use of an individual pump to hold a constant throughput in each coil would permit a number of coils in multiple in an individual pipe still. In a large unit the radiation loss per barrel is less than in a small unit, and practically the same number of men are required to operate a small unit as for a large one.

Batch distillation of crude to cylinder stocks can be carried on economically as regards fuel consumption.

With an adequate fractionating column, the crude can be split into its component parts with respect to boiling points and with the flash system a temperature of 730° F. gives a better coloured cylinder stock than 650° F. with the batch still.

In refining Pennsylvania crude, it is always desirable to split up the crude without pyrogenic decomposition, and it is possible, using a naphthene-base crude in a unit built for higher pressures and temperatures, and adding hydrogen, to obtain lubricating oils similar to those from paraffin-base oils.

With a pipe still running 2,000 bbls. of crude per day, vaporising 85 per cent., and heating to 730°F., suppose 2·7 per cent. or 54 bbls. per day are burnt with, say, 12 per cent. CO_2 and 20 per cent excess air. The combustion of this fuel in one burner takes the form of a cloud of luminous flame which conveys twice as much heat to any tube it touches than the red hot transparent gas surrounding it. The transparent gases are so hot that they tend to radiate heat and the bricks in the furnace wall radiate heat back towards these gases. The radiant bank of tubes carries oil much cooler than the flame and these tubes have a cooling effect on the gases, furnace walls, etc. Radiant heat transfer is ten times as great at 2,800° F. as at 1,000° F.

Units running on cold crude may recover heat of vaporisation sufficient to bring the oil charged to 350°F., giving a flue gas temperature of about 500°F., a thermal efficiency of 70 per cent.

Packed towers require a larger cross section area than bubble towers.

Vapour exchanges are subject to corrosion due to hydrochloric acid in the salt water found in crude oil. Whether these chlorides cause cracking at a lower temperature is an open question.

Advantages of the pipe still are :—maximum recovery of any cut, maximum fuel efficiency, large capacity in small space, maximum heat recovery and minimum cooling water required. G. S.

332. Counter Flow Still. F. E. Gilmore. U.S.P. 1,655,998. Jan. 10, 1928.

The invention provides for a still for use in refining oil, comprising a vertical shell rectangular in cross section, a plurality of sets of heating pipes horizontally disposed in vertical spaced relation in said shell, baffling material disposed between said sets of heating pipes, a hot gas inlet to the lowermost set of heating pipes, means for passing gases successively through the sets of heating pipes disposed above said lower set, a series of bubble trays disposed in the upper part of said shell, baffling material above said bubble trays, a vapour exit from the upper part of the shell, a hot oil inlet entering the shell between said bubble trays, a cold oil inlet above said hot oil inlet, a thermostatic control for the cold oil inlet, and a float controlled oil outlet near the bottom of the shell. S. W. C.

334. Purification of Liquid Hydrocarbons by Liquid Sulphur Dioxide in Counter Current. A. G. für Chemische Industrie M. B. H. E.P. 279,774. Feb. 18, 1927. Conv. (Germany). Nov. 1, 1926.

Mineral oils or oils from coal or brown coal tars are continuously treated with a counter-current of liquid sulphurous acid, the velocity of flow of the refined oil being reduced as it leaves the tower. The velocity reduction is obtained by making the cross section of the container into which the refined oil flows greater than that of the tower ; by providing a head into which the tower opens of larger cross section than the tower ; or by arranging the larger container by the side of the tower. In the latter case the acid deposited in the container may be returned to the tower by suitable means, such as a siphon. G. S.

335. Refinery Power Plant. Milon J. Trumble. U.S.P. No. 1,658,910. Feb. 14, 1928.

This patent relates to a method of utilising a large proportion of the heat now dissipated in cooling water when condensing volatile hydrocarbons after distillation. In the apparatus described, the charging stock is passed through a pipe heater and, after the addition of superheated steam, is discharged into a vaporising chamber, of special design, comprising two compartments where the volatile products are separated. This vaporising chamber consists of two drums, the vapours separating out in the first being passed on for further separation in the second. The vapours from the second chamber pass into the top of a regulating chamber, the bottom of which also receives the liquid residuum from both vaporising drums. This regulating chamber contains a float, actuating a valve, which maintains a constant level of residuum in the plant by controlling the rate at which it is released. The discharged residuum passes through a heat exchanger in which it gives up its heat to the cold oil being supplied to the pipe heater.

The vapours are led away from the top of the regulating chamber and, instead of being passed through water-cooled condensers in the usual manner, they are used to drive a turbine which is preferably of the fully-condensing type. In this manner the vapours are cooled and condensed and their heat utilised in the production of mechanical work which is preferably employed for the generation of electric power. The discharge from the turbines is mainly in the liquid phase and only requires moderate additional cooling in the normal manner before being discharged into separating tanks.

The whole plant is intended to work under a constant pressure, and any pressure fluctuations are corrected by an increase or decrease in the quantity of steam supplied, this control being effected by an automatic pressure operated valve.

It is suggested that by the general adoption of this system on a large scale very considerable quantities of electrical power could be generated, and that this power, if distributed for local industrial uses, might yield returns which would cover the running costs of the whole plant and reduce the distilled oil products to the category of a valuable bye-product of a power generating plant. E. R. B. S.

336. Construction Details of Lubricating Oil Agitators. H. L. Kauffman. *The Refiner*, 1928. 7, 2, 63.

The lubricating oil agitators described in this article are those in use at the Parco, Wyoming, refinery of Producers and Refiners Corporation. They were built by the J. G. White Engineering Corporation as part of the construction programme in building the Parco refinery. Two 1,000-barrel agitators were constructed. The lubricating oil here, in most cases a "long residuum," is treated with sulphuric acid and then neutralised and decolourised simultaneously by contacting with "100-up" mesh Florida fullers earth.

Each agitator is elevated to a height of 15 ft. and is insulated for the remainder of the height, a distance of 29 ft. The centre of the dome of the agitator is 5 ft. higher than that of the sides. The agitator is not lined with lead. Its inside diameter is 20 ft., and the distance from the bottom of the cone to the centre of the dome is 30 ft. 8 in. The cone makes a 45° slope, starting at a point 16 ft. down the side of the agitator. The diameter of the opening at the bottom of the cone is 3 ft., the opening being further reduced to 12 in. at which point the draw-off is fitted with a 12-in. standard gate valve. The cone is braced by steel trusses. An opening 1 ft. 6 in. by 2 ft. 6 in. is provided in the concrete base through which a sludge chute passes. Two doors are also provided for entrance to the base. An air-space 2 ft.

4 in. surrounds the agitator. Next is a 4-in. layer of sil-o-cel brick, while the outer layer consists of silo-tile also 4 in. thick. All concrete footings were made to extend down to good bearing on firm ground, a minimum depth of 4 ft. being specified. Concrete surfaces in contact with the ground were thoroughly coated with "Aquadam" paint. The pipe lines for each agitator include: One 6-in. charging line; one 6-in. pumping-out line; one 2-in. acid line; one 6-in. air line; one 2-in. steam-smothering line; one 3-in. high-pressure steam line to heating coils placed against the cone of the agitator; two 1¼-in. foamite solution lines; one 6-in. sewer line which connects with a 10-in. sewer line, both being buried.

Four 8-ft. diam. by 25 ft. long acid storage tanks are in use. These are provided with a 20-in. manhead opening controlled by a 1-in. stem rod leading to the top, this bottom opening being fitted with a 2-in. 30° conical outlet plug and a special forged steel outlet flange. Two 3 ft. 6 in. diameter by 9 ft. long acid blow cases are used in blowing the acid upon the oil to be treated. These are provided with a 16-in. manhead with cover; a 3-in. opening for a line that connects with the acid line leading to the agitator; a 2-in. opening for an air connection; a 2-in. opening for a filling line; and another 2-in. opening for a gauge and vent. The air compresser is a 10×17×12-in. Worthington "Laidlaw" single, horizontal, enclosed, self-oiling, steam-driven, feather valve compressor with piston steam valve and provided with a combined steam and pressure governor, steam throttle valve, lagging on steam cylinder, sight-feed lubricator or steam and air cylinders and an oil gauge for indicating oil level in crankcase. It has a displacement of 750 cub. ft. per minute at 240 r.p.m. and a volumetric efficiency of 86 per cent. Sixty-two horse-power is required to drive it, and it has a steam consumption of 28 lb. per horse-power when operating with 145 lb. steam pressure against atmospheric exhaust, 100° F. superheat, 30 lb. air pressure. S. W. C.

337. Distilling Hydrocarbon Oils. W. P. Deppé and L. L. Summers. U.S.P. 1,666,051. April 10, 1928.

An improved process related primarily to the production of motor fuel by progressively raising the temperature of crude hydrocarbon oils while maintaining all the vapours in contact with the liquid being treated in the still until a predetermined equilibrium is reached, then removing and condensing the vapours.

A suitable form of apparatus comprises three stills substantially alike and provided with oil inlet pipes. The oil to be treated enters the first still and is discharged on a suitable plate at the top of the still provided with perforations for breaking up the liquid into fine streams as it passes through the vapour chamber and falls on the uppermost of a plurality of heated plates provided with funnel-shaped openings for retaining a layer of liquid on the plates. The plates are heated by contact with steam pipes located below them. The oil passes down over a series of such plates into a heating chamber. Vapours generated are forced up through the liquid on each plate thus establishing equilibrium therewith, and successive heating stages are maintained progressively hotter from top to bottom of the still. Below the lowermost plate is provided a large chamber in the bottom of which liquid passing through the plates finally accumulates, and the upper part of which serves as a vapour chamber to which vapours from one or other of the stills may be admitted.

Vapours accumulating in the first still are conducted to the lowermost chamber of the next still, whilst the accumulated liquid is drawn from the bottom chamber of the first still and forced by a pump into the top of the

second still, where the procedure already described is followed out with progressively higher temperatures. Again, the vapours and liquid of the second still pass to a third still, from which the vapours pass to a heat exchanger and condenser giving the final fuel product.

A modified type of apparatus described incorporates the above three stills into the form of a tower still. H. S. G.

338. Fire Still and Oil Treater. F. E. Gilmore. U.S.P. 1,664,920. April 3, 1928.

An apparatus capable of fractionating oils consisting of a heating column provided with radiating fins serving as a flue for a suitable burner, and adapted to co-operate with an outer casing to form chambers to receive oil. Two or more of such heater columns may be superimposed and structurally connected by means of external flanges secured by rivets. Oil is introduced through a valved oil inlet pipe near the bottom of an upper heater section and passes down into an evaporator section provided with interior baffles in the form of conical plates. The vapours liberated escape through a tubular member and the uncondensed residue is drawn off through a valved pipe near the bottom of the lowermost evaporator compartment. H. S. G.

339. Still. F. Puening, Assr. to Koppers Co., Pennsylvania. U.S.P. 1,664,863. April 3, 1928.

A tar still adapted for continuous operation as outlined below. Within the firebrick setting of a combustion chamber is a still having a metallic bottom with a marginal flange riveted to a cylindrical vertically extending metallic heating wall that forms the fourth wall of the flue. To its upper edge is a metallic top plate having a marginal flange riveted to the upper edge of the heating wall and having a central opening with suitable removable cover. Centrally above this cover is an outlet nozzle through which vapours may be led off.

The opening may be used for connecting a rectifying tower if desired. At the upper end of the cylindrical metallic heating wall is a conduit through which vapours are returned to the still. The material inlet is located adjacent to the outlet for the heating gases, so that by counter-current flow more intense heat is imparted to the material undergoing the last stages of distillation. The combustion chamber is built so that the heating gases flow directly to the flue.

The still is divided into compartments by partitions. Cylindrical metallic baffle plates are arranged concentric to the heating wall, two such baffle plates to each compartment, and hinged so that for cleaning they may be swung out away from the walls. These baffles are so arranged that material may flow under the plates and upwardly between the heating wall and the baffles. Apertures are provided for the material to flow from one compartment to another.

In operation, heating gases flow through the space provided between the metallic heating wall and the setting coming in direct contact with the heating wall, while the material to be treated flows into the still through the inlet until all the chambers are filled and then continuously at the proper rate. The material flows from one compartment to the next in succession from the inlet towards the outlet through the apertures in the partitions. The material between the baffle plates and the heating walls flows upwardly in accordance with the thermo-siphonic principle in a thin stream as it becomes heated and the vapours flow into the space between the top level of the material and the top of the still. Thus in each compartment material flows under each baffle and upwardly between the baffle plate and the heating wall, where it is

subjected to distillation, and then flows over the upper edge of the baffle plate and downwardly in the compartment proper. This circulation results in greater productive capacity, prevents local overheating and coke formation and enables the use of a higher liquid temperature without higher shell temperature. H. S. G.

340. Designing Equipment for Chemical Treatment of Oil Distillates. J. C. Morrel and D. J. Bergman. *Chem. and Met. Eng.*, 1928, 35, 210–214.

Chemical treatment of oil distillates depends upon two fundamental principles: (1) Efficient contact between oil and reagent and (2) withdrawal of the products of reaction from the treated oil.

The orifice column has proved one of the most satisfactory devices for efficient mixing. Intimate contact of oil and reagent can be obtained by pumping them together through a series of orifice plates placed in the pipe. For most efficient action a fairly high pressure drop should be allowed across the plates (30–40 lb.) and the orifice area should be divided into several small holes. In the design of an orifice column the formulæ used are:—

$$Q = CA\sqrt{2GH}$$

where Q = Flow in cubic feet per second
C = Discharge coefficient = 0·65 for oil distillate.
A = Area of orifice in square feet.
G = 32·2 ft. per sec.
H = Head of liquid in feet

or $$Q_b = 18{\cdot}26D^2\sqrt{H}$$

where Q_b = Flow in barrels per hour
D = Diameter of orifice in inches
H = Head of liquid in feet.

Examples, and an alignment chart are given.

Baffle-plate columns serve a useful purpose as an auxiliary mixing device in conjunction with an orifice column. The efficiency of packed mixing columns is not so good as obtained with orifice columns, but better than the baffled or propellor type. Tables showing the friction drop of water flowing through 20 ft. of 4-in. pipe packed with Raschig rings are given.

Jet mixers consisting of a venturi tube or number of smaller tubes in parallel are sometimes used to mix two fluids. This type of mixer is most efficient when approximately equal quantities of liquids are being mixed, but in the chemical treatment of oil the amount of reagent is usually comparatively small. The chief disadvantage is the short time of contact of the reagent and oil. Mechanical mixers are successfully used with a number of reagents.

High velocity rotary or centrifugal pumps for direct feed of oil and reagent function as mixing devices but should always be supplemented by other mixers.

The use of blow cases as proportioning devices for feeding reagents is continued to a large extent at the present time, and can be recommended for continuous treating systems. H. S. G.

341. Refractory Problem in Oil Refining. G. Egloff and L. A. Mekler. *Oil and Gas J.*, 1928, 48, 162.

Modern high capacity tubular stills have introduced fresh refractory problems, owing to the increase in furnace temperatures and the short time between stress periods. The non-spalling quality of the refractory, that is, the ability to withstand repeated heating and cooling without disintegration, is the most important characteristic to be considered. The most suitable materials for use in the preparation of refractories for the above conditions

are those which, after firing, give a comparatively porous structure and have the minimum secondary shrinkage. A large percentage of grog, or pre-calcined material, is desirable.

High melting point is not a criterion of high quality; a much more important feature is its ability to retain its shape and rigidity above the operating temperatures of the tube still, about 2000° F.

A refractory for refinery use should run true to size and have as sharply defined edges as possible, and the minimum of cement should be used in laying. It is found that a refractory made in a tunnel kiln will stand up better than one made in a periodic kiln. In a properly designed still there is no need for super-refractories such as silicon carbide, etc.

Numerous tests are now being made with insulating materials. They are lighter in weight and have approximately one-quarter the heat conductivity of firebrick. They are porous and thus liable to disintegration by action of the combustion gases unless protected by a wash of refractory cement which vitrifies under the operating temperature of the furnace. The outside surface is usually covered with mineral wall board or weather-proofing to protect it from the elements. Most of the weaknesses that the conventional furnace structure develops is due to strains that would be absent in a tube still built up of smaller thicknesses of uniform insulating refractory. H. S. G.

342. Apparatus for Treating and Combining Hydrocarbon Liquids and Gases. N. H. Colwell. U.S.P. 1,668,104, May 1, 1928.

The object of this invention is to treat and combine by conversion with or without heat through the application of pressure hydrocarbon liquids and gases into a composite product of stable consistency and predetermined characteristics. The products to be treated are introduced through pipes under pressure into an impact chamber where the columns of products are caused to meet at an angle impinging on each other with considerable impact. The product is divided and drawn into a pair of pumps, thus being subjected to a churning treatment, and forced into an impact chamber, the two streams meeting at an angle with considerable impact. From this chamber the product in a single stream is forced through one or more orifice plates into a manifold disposed in a stable pressure tank and caused under high pressure to pass through numerous mechanical vaporising discharges which completely vaporise the product under treatment. The product finally condenses in a pressure tank and becomes a composite product homogeneous in character and of a predetermined specification. The process is adaptable to operate with or without heat. H. S. G.

343. Improvements in Centrifugal Separators. F. A. G. Noel and E. W. Baily. E.P. 288,746, Jan. 24, 1927.

An improvement in centrifugal machines suitable for separating two fluids without aeration, consisting in conducting the fluid into a rotating annular chamber containing a stationary collecting device with an annular orifice and causing it to flow radially inward and upward through the collecting device and a stationary annular duct into a chamber provided with a discharge pipe. In practice the bowl and spindle are constructed on the usual lines, and fluid is fed into the machine through a stationary inlet pipe located on the axis of the machine and an annular chamber is formed about this inlet pipe in the cover secured to the bowl. Treated fluid rises into this chamber and is removed through a stationary discharge device suitably supported above the machine and projecting into the annular chamber. The discharge device consists of two concentric sleeves spaced apart to form an annular

vertical passage with the lower ends flanged and also spaced to form an annular inlet opening of large area leading from the chamber in the cover to the vertical passage between the sleeves. The outer sleeve carries a delivery port and delivery pipe so arranged that the fluid is conducted from the annular chamber in the drum cover, through the annular inlet opening between the flanges, then through the vertical passage between the sleeves whence it is discharged through the delivery pipe in a continuous stream, without any entrainment of air. Baffles of suitable form and disposition are arranged in the annular opening between the flanges and in the annular passage between the sleeves to check the rotation of the fluid without formation of eddies. H. S. G.

344. Process of Treating Gases, Vapours or Liquids. Chemische Werke Carbon, Gesellschaft m.b.H. E.P. 266,673, Nov. 29, 1926. Conv., Feb. 24, 1926.

According to the present invention gases, vapours or liquids are treated in order to remove impurities or constituents thereof, to carry out catalytic reactions between them or to mix them, with highly active carbon blocks, characterised by an extraordinarily high mechanical strength and hardness, the latter being at least 10 on the Shore scale as determined with the sclerescope. The process for the manufacture of such carbon blocks is described in E.P. 266,674. H. S. G.

345. Method of Refining Heavy Mineral Oils by Means of Sulphur Dioxide in a Continuous Operation. Edeleanu, Gross and Jodeck. U.S.P. 1,666,560. April 17, 1928.

This patent is a continuation in part of an application filed September 13, 1924, Serial No. 737,542.

Heavy mineral oil and liquid SO_2 are delivered into the top of an enclosed tank fitted with a stirring device and the emulsion of oil and extracting medium passes from the base of the tank, through a pipe into another tank, the pipe terminating in an annular orifice of gradually increasing area, the end of the orifice being horizontal. Thus the mixture is delivered in a radial direction at a slow speed. This causes settling of the emulsion into a layer of refined oil which is drawn off at the top of the tank and one of SO_2 extract which is drawn off at the bottom.

The cost of refining by this process can be materially decreased by connecting a number of these washers in series for counter current washing. A. R. S.

Engineering.

346. Report on Heavy Oil Engine Working Costs (1926-1927). Diesel Engine Users' Association, 1928.

The report of the Diesel Engine Users' Association on Heavy Oil Engine Working Costs during 1926-1927 was submitted at a meeting of the Association on February 24, 1928.

Details are given of plants and costs at 53 stations, employing 205 engines and generating 74,912,880 units. These 53 plants consumed 24,208 tons of fuel oil and 126,451 gallons of lubricating oil. The 41 home stations dealt with employed 142 engines and generated 44,060,256 units, with an average of 2,155 engine hours run. The total fuel oil used was 14,182 tons, or 0·721 lb. per unit generated, and lubricating oil 74,210 gallons, or 0·00168 gallon

per unit. The average engine cost per unit generated was 0·713d. as compared with 0·799d. for 1925-26.

Details are given for the working of 12 overseas stations which generated 30,852,624 units, using 63 engines and operating on an average of 2,433 hours per engine. The fuel oil consumption was 10,026 tons, an average of 0·728 lb. per unit, and the consumption of lubricating oil was 52,241 gallons, an average of 0·00169 gallons per unit. The average engine cost per unit generated was 0·665d. as against 0·753d. for the preceding year.

The fall in the average engine costs per unit generated by the home stations is due mainly to increased capacity and output of the stations and it is interesting to compare the changes in each item during the past five years, as follows.

	1926–27.		1922–23.	
Output (millions of units)	44·1		21·7	
Kilowatts installed (thousands)	33·3		16·6	
Average engine costs per U.G.	Pence.	%	Pence.	%
Fuel	0·353	49·49	0·367	38·86
Lubricating oil	0·065	9·11	0·071	7·53
Water and stores	0·021	2·94	0·022	2·34
Wages (running)	0·167	23·42	0·296	31·36
Repairs	0·107	15·04	0·188	19·91
	0·713	100·00	0·944	100·00

Taking the gross calorific value of the fuel used at 19,500 B.Th.U. per lb., the heat consumption per unit generated over the whole 53 stations averaged 14,110 B.Th.U., an overall thermal efficiency of 24.2 per cent.

Little special information regarding fuel oil has been contributed during the year but a useful note was received from the Worthing station. Here the fuel used included 380 tons of oil of a viscosity of 450 secs. at 60° F., 315 at 70° F. and 145 at 100° F., normally classed as a 300 seconds oil. On account of its alleged unsuitability for Diesel engines a number of tests were made, one of which gave :—

Sp. gr.	0·921
Flash-point (open)	221° F.
Water	0·5 per cent.
Coke	6·55 ,, ,,
Ash	0·08 ,, ,,
Asphaltum	4·75 ,, ,,
(Hard asphaltum)	3·5 ,, ,,
Calorific value	18,081 B.Th.U. per lb.

The behaviour of this fuel, particularly as to wear, is being studied and recent engine examinations do not indicate any accelerations in rate of wear. Possibly centrifuging would be desirable and experiments are being made to confirm this. A long burning period is necessary and pulverisers require adjustment to prevent restriction. G. S.

347. DISTILLING DEVICE FOR LUBRICATING SYSTEM OF INTERNAL COMBUSTION ENGINES. G. H. Greenhalgh, Assr. to E. J. Sweetland. U.S.P. 1,668,013. May 1, 1928.

A distilling device which can easily be applied to the oiling system of an internal combustion engine capable of separating water and light hydrocarbons from the lubricating oil. The distilling device is mounted upon an exhaust manifold from which the exhaust gases are utilised for heating purposes, and comprises an inner spherical vessel and outer spherical vessel arranged

concentrically. The inner vessel is provided with inlet and outlet openings for the oil to be treated which extend in the form of pipes through the outer and inner vessels and space between them. An outlet opening in the top of the inner vessel provides a means for carrying off distilled vapour. The outer vessel has inlet and outlet openings capable of connection to the exhaust manifold for conveying exhaust gases. A controlling device is so arranged that it moves in synchronism with the throttle thereby regulating the admission of exhaust gases. The wider the throttle the less the amount of exhaust gases passed through. In this way excessive heating is prevented. The distilling device may be arranged to receive the whole or a portion of the oil in circulation in the lubricating system of the engine, and operates by vaporising the major portion of the lighter hydrocarbons and water in the oil. The vapours may be carried off to a suitable point and either condensed or allowed to escape as desired. H. S. G.

Antidetonation.

348. KNOCKING CHARACTERISTICS OF GASOLINES. *Oil and Gas J.*, 26, **39**, p. 140.

The method of rating used by the Bureau of Standards in these tests was the variation of throttle opening in a single cylinder high compression liberty engine; for each fuel fed to the engine the throttle is opened until the standard intensity of pinking in reached, the horse power developed at that setting is taken as the knock rating of that particular fuel.

Various refineries submitted gasolines typical of current refining production. (S.-R. denotes straight-run; Cr. denotes cracked; Bl. S. denotes blending stocks.) Knock ratings and benzol equivalents were as follows:—

	K. R.	Benzol Equivalent.
Pennsylvania, S.-R.	100	0
Amarillo, S.-R. Oklahoma, S.-R.	105·6	15
Ohio, S.-R. Salt Creek, S.-R.	109·6	20
Salt Creek, Cr. Commercial, Pa. Amarillo, Cr.	114·5	25
Commercial, N.J.	123·6	35
Smackover, Bl. S. Spindletop, S.-R.	131·5	
Los Angeles, S.-R.	136·0—140·5	
Spindletop, Cr.	146	

Representative samples were made up from non-premium fuels from filling stations in various parts of the U.S.A., so that the six fuels obtained were average samples from six districts. Knock ratings and benzol equivalents were as follows:—

	K. R.	Benzol Equivalents.
Chicago	100	0
Denver	101·5	
New York and New England	103·0	10
Texas	105·0	15
California	118·4	30

W. N. H.

349. AUTOXIDATION DURING SLOW COMBUSTION. E. W. J. Mardles. *J.C.S.*, April, 1928.

Continuing the work detailed in "Engineering," Feb. 4, 1927, hexane-air mixtures were passed through tubes heated to 500° C., 350° C., and 300° C. The hot gases were passed into slightly acidified potassium iodide solution and the liberated iodine was titrated with $\frac{N}{50}$ sodium thiosulphate solution. Only small quantities of "active" oxygen were found when rate of passage of the gas mixture was slow; with increased mixture velocity, much larger yields were obtained, particularly if the hexane was introduced as a fine spray. Rapid chilling of the products of combustion failed to yield any peroxides.

The formation of "active" oxygen in a 15 per cent. hexane-air mixture is inhibited by substances known to possess anti-knock properties; for a 25 second period of heating, results were obtained as follows:—

Substance added to mixture and approx. proportion.	Effect on amount of "active" oxygen produced.
Ethyl alcohol, 5 per cent.	Marked decrease.
n-butyl alcohol, 2 per cent...	Slight decrease.
Benzene and toluene, 5 per cent.	Moderate decrease.
Aniline, 2 per cent.	Marked decrease.
Phenol and m. cresol	" "
Carbon monoxide, 10 per cent.	No "active" oxygen observed.
Lead tetraethyl, ·2 per cent.	" "
Iron carbonyl, ·1 per cent.	" "

With the last three substances the yield of aldehydes in the products of combustion was substantially reduced.

Where benzene vapour was added to hexane-air mixture and heated to 500° C., the products of combustion, on being passed through bromine water, produced a precipitate of tri-bromophenol, showing that benzene had been oxidised; at 350° C. oxidation of aniline in an aniline-hexane-air mixture was observed, although by itself an aniline-air mixture is oxidised to a similar extent only on heating to 500° C.

When a spray of water was injected into a hexane-air mixture, the temperature of initial combustion was considerably raised, a similar effect being obtained with a 60 per cent. hydrogen peroxide solution.

The effect of peroxides in inducing detonation was found to be as follows in the Ricardo E. 35 variable compression engine

	H.U.C.R.
Ethyl ether 60% Ethyl alcohol 40% by vol.	4·9
" " " +5% water	5·4
" " " +50% of 60% H_2O_2 soln. .	5·2
Petrol	4·9
" +0·5% (wt.) methyl ethyl ketone peroxide	4·2
" +2% (wt.) ethyl hydrogen peroxide	4·1

The influence of hydrogen peroxide on the temperature of initial combustion (liquid sprayed into the tube) was found to be as follows:—

	T.I.C.
Ethyl alcohol alone	570° C.
" " +10% (vol.) of water	575° C.
" " +10% (vol.) of 60% H_2O_2 soln.	570° C.
n-butyl alcohol	545° C.
" " +10% (vol.) of water	559° C.
" " +10% (vol.) of 60% H_2O_2 soln.	560° C.

This shows that the hydrogen peroxide found in exhaust gases is not in itself a detonation inducer, but must be the decomposition product of much more active and unstable peroxides formed at first.

Using the Moore spontaneous ignition temperature apparatus the following results were found for the influence of detonation inducers on the S.I.T.

Substance.	Detonation Inducer added.	S.I.T.
n-butyl alcohol ..	Nitrogen peroxide 0% (vol.) ..	590°
	" " 2·5% ..	355°
	" " 5%	235°
	Benzoyl peroxide % (wt.)—	
	0	585°
	2	560°
	Iso amyl nitrite % (vol.)—	
	0	590°
	2	552°
	5	545°
	10	518°
	20	455°
m. xylene	Nitrogen peroxide % (vol.)—	
	0	635°
	2·5	430°
	Benzoyl peroxide % (wt.)—	
	0	629°
	2	613°
	Iso amyl nitrite % (vol.)—	
	0	635°
	2	567°
	5	535°
	10	507°
Ethyl alcohol.. ..	Benzoyl peroxide % (wt.)—	
	0	570°
	2	547°

Combustion at low temperatures was also studied by the bulb method. Two similar bulbs were heated in a well-stirred thermostat, one being filled with dry air, the other with the hydrocarbon air mixture to be tested. The increased pressure due to heating was balanced by a differential arrangement, a toluene manometer measuring the pressure increase due solely to combustion.

A 28 per cent. ether-72 per cent. air mixture showed a very slow combustion in the bulb at temperatures between 120° and 175° C.; between 175° and 180° combustion took place very rapidly, until the reaction is complete. A 12 per cent. ether-88 per cent. air mixture reacted rapidly at 195°–205° C., and a 6 per cent. ether-94 per cent. air mixture at 215° C.

A mixture of 15 per cent. hexane-85 per cent. air showed rapid combustion between 255° and 260°; when 5 per cent. of lead tetraethyl was present the pressure due to combustion rose steadily between 220° and 320°.

A mixture of ether and air plus 5 per cent. lead tetraethyl showed fairly rapid combustion at 150°–160°, this became rapid at 220°, while in absence of the lead tetraethyl, combustion is rapid at 175°–180°; this confirms previous results obtained by the " tube " method. W. N. H.

350. Methods of Measuring Detonation. N. MacCoull. *Oil and Gas J.* May 10, 1928.

Samples of six fuels for internal combustion engines were sent to ten different laboratories, the workers in each of which measured the pinking properties of each fuel.

Fuels A.K. and D.K. had an end-point on distillation of 436° F.; B.K. consisted of A.K. plus 3·4 c.c. lead tetraethyl per gallon; C.K. consisted of 50 per cent. A.K. and 50 per cent. benzol; E.K. had A.P.I. gravity 60·2, end-point 392° F.; F.K. had A.P.I. gravity 52·9, end-point 390° F.

Three laboratories employed variable compression engines, the remainder induced pinking by variations in the throttle opening; three laboratories estimated the intensity of the pinking by the bouncing pin, the remainder relied on an audibility method. Each laboratory employed as a fuel of reference some special petroleum spirit.

Very large divergences in the estimations of the laboratories were found. These are discussed in detail. The most satisfactory correlation was found between the three laboratories using variable compression engines. The following conclusions are drawn:—

(1) There is great need for further development of anti-knock measurements.

(2) Certain elements of technique such as the mixture ratio and spark setting should be standardised at once.

(3) Intensive study should be made of the fundamental methods in use for the direct control of detonation in test engines, such as the compression ratio, manifold vacuum, etc.

(4) Measurement of anti-knock values in terms of the mixtures of a reference fuel with benzol or tetraethyl lead is apt to be very misleading if the peculiarities of such scales are not thoroughly understood.

(5) The data given in the survey indicate that no great difference in the results probably exists whether the detonation point is determined by audibility, the maximum power or the bouncing pin.

(6) The size and type of engine used apparently does not affect the results.

(7) Eventually a reference fuel of low anti-knock value and one of high anti-knock value should be standardised.

The following recommendations are made:—

(1) A standard scale for measuring anti-knock values should have some direct connection with the compression ratio.

(2) If all laboratories measured anti-knock values in terms of percentage of change in the compression ratio for variable compression engines or of manifold vacuum for fixed compression engines, the data obtained from any engine might be converted into those obtained from any other engine by the use of a single multiplying constant. W. N. H.

351. Flame Movement in Gaseous Explosive Mixtures. Part I. O. C. de C. Ellis. *Fuel*, May, 1928.

General considerations of conditions obtaining in explosive mixtures lead the author to the following conclusions: When an explosive mixture is ignited at the centre of a spherical vessel, the pressure is communicated along a falling gradient and is therefore accompanied by a surge in the gas affected. There is a finite period between ignition and the moment at which pressure is increased at any point at a finite distance from the point of ignition. The pressure is generated from the expanding layer of gas adjacent to the flame surface and within it. It presses outwards and inwards. Outwards, it displaces the flame-surface increasing the total volume of the flame gases and complementarily compressing the unburned gas surrounding the flame; inwards it compresses the flame gases. The first junction is the more marked, while the volume of the flame is comparatively small, but as the flame grows its effect gradually decreases. A time comes when the pressure generated is distributed equally outwards and inwards. This happens when the volume of the flame is approximately half the volume of the sphere, the mean pressure

in the flame gases being approximately equal to the mean pressure in the unburned gas surrounding the flame. At this critical moment the second junction is increasedly marked as the volume of unburned gas decreases. There is therefore a gradual arrest of the distension of the flame which reaches a critical point when the volume of the flame is approximately half that of the explosion vessel—that is, with a sphere, when the diameter of the flame is approximately four-fifths that of the explosion vessel.

This arrest would not occur if there were no increase in pressure in the unburned gas surrounding the flame—that is, if the gas were not enclosed. In the example considered, the pressure upon the flame is everywhere uniform. Other examples of the effect of the pressure in the unburned gas upon the flame-surface are the translation and change of shape due to upthrust and lifting pressure; the translation and change of shape due to a symmetric ignition or to the non-spherical form of the explosion vessel; and, lastly, the adhesion of a flame, having an incomplete luminous surface, to the wall of an explosion vessel. W. N. H.

352. PREPARATION OF LEAD TETRAETHYL. Y. Tanaka and T. Kuwata. Rept. of Aeronaut. Research Inst., Tokyo Imp. Univ., No. 29, December, 1927.

Lead tetraethyl may be prepared by the reaction of ethyl magnesium iodide or bromide (Grignard's reagent) with lead chloride. It is known that ethyl iodide or ethyl bromide is a good starting material, whereas ethyl chloride cannot be used on account of weak activity.

The authors have found that an ethereal solution containing ethyl iodide (or bromide) and iodine is a suitable catalyser for the reaction between ethyl chloride and magnesium. When this solution is added to magnesium, ethyl magnesium iodide, $C_2H_5Mg.I.$, is instanteously formed and accelerates catalytically the reaction between ethyl chloride and magnesium. The crude lead tetraethyl may be readily prepared by the reaction of ethyl magnesium chloride and lead chloride.

The crude lead tetraethyl, containing a considerable portion of unsaturated lead triethyl, is treated with air or oxygen in the presence of dilute hydrochloric acid, the lead triethyl being converted into a crystalline mass of triethyl lead chloride $(C_2H_5)_3PbCl$. The latter is then transformed into lead tetraethyl by the action of ethyl magnesium chloride, with a yield of 95 per cent. of the theoretical. The total yield of lead tetraethyl to the amount of ethyl chloride used was about 74 per cent. of the theoretical.

The pure lead tetraethyl had a sp. gr. $\left(d\ \frac{18°}{4°}\ \text{vac}\right)$ of 1·660, refractive index $\left(n\ \frac{18°}{D}\right)$ of 1·5206, and a boiling point of 81·6° to 82° C. at 11 mm. or 85·4° to 85·7° C. at 13 mm. It decomposes at about 400° C., separating metallic lead. Other properties were also investigated. G. S.

Cracking.

353. CRACKING OR HYDROGENATION? Dr. M. Naphtali, Berlin-Wilmendorf. *Petr. Zeit.*, Vol. 24, No. 14, p. 611.

The principles of the most highly-developed cracking processes as elaborated by Leslie and Potthoff for American oils, Auld and Dunstan, and Ssachanow for Russian oils are described briefly. According to *Leslie and Potthoff* the appropriate cracking temperature is 426° C., and the process is promoted catalytically by the walls of the metallic vessel. The formation of gasoline

is expressed by the equation $K = x/A.t$ where the percentage content of gasoline formed $A = (100 - x)$, t is the time in minutes and k a reaction constant. The process is endothermic and requires 500 calls./gm. gasoline produced. *Auld and Dunstan* (cf. *Brenn. Chem.*, 18, 803, 1926) state that the pressure must be high enough to maintain the oil to be cracked in the liquid form and to maintain the quantity of undissolved gaseous reaction products as low as possible. They utilise a special apparatus and emphasise the importance of the time factor. For every oil there is a definite narrow temperature range in which cracking begins, and the quantity decomposing increases rapidly whilst the velocity of reaction is almost doubled for every 10° increase in temperature. There is a definite time factor controlling the formation of light benzine, which is a function of the temperature.

The results of *Ssachanow and Tilitschew* show that the velocity attained with the maximum yield of benzine from distillates rich in paraffin amounts

at—375°	400°	425°	450°	475°	500°
to—45h.	9h.	1½h.	12′	2′	20″

respectively and the velocity of decomposition increases usually with increasing specific gravity of the fraction. A temperature increase of 10° causes a doubling of the velocity of cracking. The olefine content of the cracked benzine decreases with increase of pressure and those olefines formed give rise to other condensation products (not naphthene rings).

With regard to the technical application of cracking, the liquid phase cracking is preferable to the vapour phase process on account of higher yields and better products.

One of the more important of the liquid phase processes is the *Burton Process* of the Standard Oil Co., in which gas oil and heating oil are distilled in special boilers under 4–5 atmospheres excess pressure. Other processes are the *Jenkin's Process*, which is applicable to crude oil, topped oil, gas oil, heating oil and suitable mixtures; the "*Tube and Tank*" *Process* developed by Ellis of the Standard Oil Co., in which the oil, previously brought to cracking temperature in a heating tube, is driven slowly through a reaction chamber and the vapours formed finally led through a dephlegmator from which the condensed portions return to the cracking coils. Also the *Dubb's Process*, by which 80 per cent. of the crude oil can be converted into cracked benzine by heating the oils to above 400° C. and at 7–10 atmospheres pressure; *Dr. Erwin Blümer's Process*, in which the crude material is pumped into an autoclave filled with liquid lead charged with Rasching rings and distributing substances; 60–70 per cent. of the crude is thereby converted into benzine at 40 atmospheres pressure; *Melamid's Process* is similar to the latter process, but a liquid metal is used—viz., tin or tin alloy which acts as a heat transmitter and a catalyst in the presence of hydrogen.

Regarding catalytic processes in the absence of H_2, the conversion of crude oil or distillates with aluminium chloride has been utilised by the Gulf Refining Co. Also a new process in which the liquefaction of coal is enhanced by catalytic and electrical effects has been attempted by *Krauch of the I.G. firm.*

Contrary to the Bergius process, which causes the hydrogenation of coal without catalysts—since the usual catalysts are subject to rapid poisoning under the influence of coal gas containing sulphur—the *I.G. die industry* has tried almost all conceivable substances and combinations of substances as catalysts in its hydrogenation processes. All types of coal from peat to anthracite coals, tar and mineral oils, have been submitted to hydrogenation. Fe, Mn, Zn, Al, Mo, ZnO, MgO, active carbon, Al $(OH)_3$, $CeCO_3$, Al_2O_3, Mo and its compounds and various other catalysts have been utilised. These are mentioned in respect to the various processes and the English patents enumerated. W. S. C.

354. METHOD AND APPARATUS FOR CONTINUOUSLY CONDENSING OIL-CRACKING STILL VAPOURS. Richard Fleming. U.S.P. No. 1,655,053. Jan. 3, 1928.

The broad subject-matter of this invention is disclosed in Patents Nos. 1,325,668 and 1,394,987 of Dec. 23, 1919, and Oct. 25, 1921, wherein the vapour line from a high pressure cracking still leads to a nozzle constructed to maintain the desired cracking pressure and also to discharge the vapours continuously at high velocity into water. This is a continuation in part of the above patents.

The invention effects improvements in the condenser by providing means for varying the nozzle aperture by means of a needle and also arranges water cooling for the needle.

The condenser is arranged in the form of a valve, the still vapours entering around the needle. The vapours are discharged through an annular orifice, the area of which may be varied by moving the tapered needle. Water is admitted concentrically with the vapours through the hollow needle and also through another annular space surrounding the vapour orifice. The vapours are discharged at very high velocity and they and the two water streams pass into a species of venturi tube where intimate contact, and consequently rapid cooling, is assured. The mixture passes from the condenser to suitable settling tanks or other means of separation.

The water may or may not be supplied under pressure as it is found that the ejector action of the gases will lift the cooling water an appreciable height.

It is suggested that the extremely rapid cooling realised may improve the performance of a cracking still by allowing very little time for dissociation of molecules into gases and unsaturated hydrocarbons.

A purifying operation may be combined with condensation by the mixing of suitable purifying agents with the cooling water. E. R. B. S.

355. APPARATUS FOR TREATING OIL. G. D. Sims. U.S.P. 1,658,420, 1928. Appl., March 10, 1927.

The height of liquid in a cracking still or expansion chamber is indicated by means of a gauge glass, one end of which is connected through a cooling coil to the still or chamber and the other to the pressure distillate receiver.

H. G. S.

356. CONTROL OF PYROLYTIC CONVERSION. J. R. Carringer. U.S.P. 1,656,724. Jan. 17, 1928.

In a plant consisting of a cracking coil and a reaction chamber wherein the heat necessary for the reaction is imparted to the oil in the coil and the process is completed in the reaction chamber, which is protected from loss of heat by insulation or by waste flue gases, regulation of the temperature of the oil entering the reaction chamber is obtained by interposing, between the coil and the chamber, an air-cooled coil. The latter coil is housed in a tower composed of a shell, having at its upper end outlets for air and, at its lower end, inlets with adjustable dampers. A by-pass line is conducted round this structure to allow the oil to pass either directly to the reaction chamber or part of the oil only may be by-passed. Other cooling media may be used, such as steam, water or oil, these being usable when and to the extent desired. R. P.

357. CRACKING OILS. F. Puening. U.S.P. 1,664,263. March 27, 1928.

The apparatus is intended to alleviate the danger due to the deposition of coke in cracking stills. The method consists in subjecting an annular column

of oil to a cracking heat, while effecting a rapid circulation of the oil by thermo-syphonic action and at the same time mechanically removing the carbon from the walls surrounding the column of oil.

The apparatus consists of a vertical still having its extremities situated outside the heating zone. An interior shell, open at top and bottom, provides the annular space through which the oil circulates by virtue of the heat imparted. Steel wire brushes are attached to the inner shell, which is rotatable by means of a central shaft, and these brushes remove the carbon as it is formed on the heated surface of the still. The brushes are attached by means of a steel band, which acts as a spring to maintain contact between the brushes and the shell of the still.

The annular space is purposely made small and the central duct large so that the velocity of the oil is high, in contact with the heated surface. Thus the settling out of the carbon is made difficult in the hot region, but is carried upward and into the central duct where it settles down easily. R. P.

358. Flash System Aids Cracking Operation. C. O. Willson. *Oil and Gas J.*, April 12, 1928.

An old battery of Dubb's units has been rebuilt, incorporating the flash system of operation. This method has recently been made available to refiners and has increased the gasoline yield with small deposition of coke and production of a commercial grade of fuel oil.

The problem was to modernise a battery of four Dubb's units which had been in operation for a number of years. These units had the small 10 ft. by 10 ft. reactions chambers without hot oil pumps and the results obtained latterly were not comparable with those obtainable with modern plants.

The new method of operation increased the capacity so that all four units were not required to handle the through-put. One unit was therefore used as a reducing still, and as now operated one unit furnishes the gas oil charging stock for three cracking units. The capacity of the reducing still was enlarged by increasing the number of heating tubes from 50 to 82. The oil flows through these and is ejected into the 10 ft. by 10 ft. flash chamber, where 90 per cent. is volatilised and passes into the dephlegmator. The residuum is drawn off from the chamber and is circulated as fuel, supplying enough for the reducing still and two cracking units. In the dephlegmator the vapours are condensed with the exception of about 15 per cent., which passes overhead, giving a naphtha cut which is re-run along with the pressure distillate from the cracking units.

The gas oil accumulates in a stock tank whence it is pumped into the cracking section of the plant. The fresh oil is mixed with hot recycle oil from the cracking plant and pumped at about 500° F. to the dephlegmator. The hot vapours from the reaction chamber of the cracking plant further preheat the oil, then it passes into the tubular heater, where the temperature rises to about 885° F. The cracked oil emerges into a 5 ft. by 40 ft. reaction chamber at a pressure of 225 lbs./sq. in., and the liquid which remains in this chamber is released into a flash still. A small percentage of fuel oil is taken from the bottom of the flash still, whilst the vapours pass to the mixing drum, where they meet the first charging stock as already explained.

The vapours from the reaction chamber pass to the dephlegmator, which separates the pressure distillate from heavier recycle oil.

Up to date there is not a great difference in the percentage yield of gasoline from the new method, 49 per cent., as compared with 46 per cent., of 400° F. E.P. gasoline, but the advantage lies in the greater capacity and increased value of the residual products. With the old method the units operated on a four-day cycle, three days operating and one cleaning. One of the new units

has just finished a 30-day period on stream. Based on the monthly operation, therefore, two units will handle 35,000 bals of charging stock, as compared with 20,000 by the old method.

By the old method 13 per cent. of fuel was obtained, but this was not a marketable product, whereas by the new method 32 per cent. of commercial-quality low cold test fuel is available.

Also, by the old method, 7 to 8 per cent. of coke was made, whilst less than 1 per cent. is produced by the new method of working.

Moreover, the anti-knock value of the spirit produced is higher than the same produced by the old method. In the old method a pressure of only 180 lbs./sq. in. was used, whereas in the new plant 225 lbs./sq. in. is attained.

R. P.

359. Art of Cracking Hydrocarbons. G. H. Taber, Jr. U.S.P. 1,663,868 March 27, 1928.

The gases and vapours from a cracking plant after passing through a reflux tower and a condenser are separated into liquid and gaseous products. The gases are further treated for recovery of light products by passing them through a tower of conventional construction. A suitable absorbent, such as mineral seal or light straw oil, is circulated in a cycle to the scrubbing tower, thence to a recovery still, then back to tower, after the absorbed constituents have been removed. The gases, after this treatment, pass into a second scrubber, wherein they are treated with fresh charging stock, for example, gas oil, which is afterwards fed into the cracking plant.

The absorbent may be charged to the cracking still directly, or after passing through a reflux tower in contact with the hot vapours escaping from the cracking still.

R. P.

360. Improvements Relating to the Treatment of Heavy Hydrocarbons for the Production of Lighter Hydrocarbons. C. Arnold (Standard Development Co., U.S.A.). B.P. 286,917. March 15, 1928.

The process is a continuous low temperature method for cracking, wherein the raw oil is heated to a decomposing temperature, whilst being rapidly forced under pressure through a heating coil at such a rate that no substantial decomposition takes place. The heated oil is continuously passed through a cracking zone—a series of chambers or converters—without distillation and at such a slow speed that sufficient time is given for the desired degree of cracking to take place. Sufficient heat is applied to the cracking chambers to prevent loss of heat only, without raising the oil temperature.

The apparatus consists of a coil of tubing situated within a furnace for preheating the oil to the desired cracking temperature. This coil is connected to a battery of vertical cracking retorts which are also placed in heating chambers. The heating of these retorts can be accomplished either by heating them outside or inside, with fire tubes, but preferably in such a manner that the temperature of the heating agent is kept close to the temperature of the oil, so that overheating is avoided. Rapidly circulating flue gases will achieve this requirement.

Whilst operating, any of the cracking chambers may be cut out for cleaning without disturbing the continuity of the process. Pipe lines carry away the cracked oil from the top of one converter to the bottom of the next, until it has passed through the series in use.

Finally, the oil passes into a vapouriser, where the pressure is released and the light constituents vaporise and pass through the usual condenser and gas separator.

R. P.

361. Oxidation of Hydrocarbon Oils. E. E. Reid. U.S.P. 1,663,523. March 20, 1928.

The process relates to the treatment of heavy petroleum oils, and particularly high boiling paraffin hydrocarbons, to an intensive oxidation at an elevated temperature, with the object of improving the efficiency of cracking high boiling fractions for the production of gasoline. Also, to provide a process of oxidizing difficultly oxidizable hydrocarbons which shall be so controllable as to permit the recovery of the relatively unstable oxygen derivatives. By means of this invention, the air used for oxidation is comminuted and disseminated through the oil in an extreme degree of subdivision. In this way the speed of reaction is multiplied many times as compared with a reaction carried out with ordinary agitation or stirring. On account of the rapid oxidation, the process may be carried out at temperatures considerably lower than those which have previously been considered practicable.

The apparatus specified consists of a still adapted to withstand a high pressure and provided with a Witt stirrer (described in *Berickte der Deut. Chem. Gesell.*, vol. 26, p. 1696), wherein the air is introduced through the stirrer arms. With a stirrer of this type, the oil is drawn into the stirrer and ejected at high velocity through the body of oil, entraining air in its passage. Instead of rotating the stirrer at the usual speed of several hundred revolutions per minute, the stirrer is rotated at several thousand revolutions per minute.

The temperature of cracking depends on the quality of the raw oil and the character of the cracked distillate required. For an oil from which gasoline and kerosine have been distilled a temperature of about 370° to 425° C. may be used, and a pressure of about 300 lbs. is applied, but the pressure may vary, as it depends on the volatility of the oil, but in general sufficient pressure is used to maintain most of the oil in the liquid phase.

After the oxidation has started, external heat is withdrawn and the progress of the reaction is regulated by the rate of admission of air. As the action proceeds gasoline is formed along with carbon monoxide, carbon dioxide and water, which pass through a condenser. These products being in the vapour phase, are immediately removed, and permit the process to be carried on indefinitely without the formation of solid carbon in the still. R. P.

362. Apparatus for Removing Coke from Stills. L. V. Robbins. U.S.P. 1,666,865. April 17, 1928.

The top of the still is provided with a large manhole, preferable equal in area to the still top. The actual coke removing apparatus is set into the bottom of the still before commencing the run. This apparatus consists of a series of flexibly connected strips of suitable material, such as boiler plate. Each of the strips is fitted with lateral teeth and attached to the last strip is a drag, running on guides, which is made of I beams and connecting angle irons. After the run the flexible strips are drawn out by attachment of the connecting chain through the manhole to a windlass. It is claimed that withdrawal of the complete apparatus clears the coke from the still and that the coke is easily removed from the drag. A. R. S.

Oilfield Technology.

363. Studying Corroding Action of Soils. H. D. Holler. *The Oil and Gas Journal*. Vol. 26, No. 32, p.45.

There are four ways in which oxygen may gain access to an underground iron pipe to produce corrosion.

(1) Air may penetrate the soil by gaseous diffusion, and so long as the soil be of fairly loose texture a sufficient volume to account for normal corrosion could be obtained in this way.

(2) Air dissolved in water may be carried to the pipe in solution. The quantity of water required to carry a sufficient amount of air for normal corrosion would appear too large for this to account for more than a small proportion of corrosion encountered.

(3) Oxygen may be obtained from the decomposition of water and would require a very small amount of water to produce normal corrosion.

(4) Metallic and non-metallic oxides present in the soil may be decomposed to provide oxygen by the process of "base exchange." There is no evidence to hand on this point.

It may be taken that where a pipe loses weight at the rate of 1 oz. per sq. ft. per year (about 3·3 gms. per 100 sq. cms.) the rate of corrosion is serious especially if marked pitting occurs.

To corrode 100 sq. cms. of iron at the rate of 3·3 gms. per year by the decomposition of water would liberate about 3 to 4 cu. cms. of hydrogen per day and experiments have been made to investigate the possibility and extent of this reaction.

Pulverised cast iron which would pass through a 50-mesh sieve was mixed with the soil to be tested, 10 gms. of each being taken, and the mixture placed in a graduated test tube filled with water. The test tube with its contents was inverted under water and left for reaction to take place. In most cases action began promptly and hydrogen began to collect in the top of the tube, but with some soils there was a delay of several days before gas started to form. Different soils had widely different ranges of activity, the volume after twenty-five days varying from 12 cc. to 286 cc. Repeat tests on several typical soils checked usually to within 1 or 2 per cent., so that the volume of hydrogen liberated may be taken as a measure of the corrosive action of various soils under certain conditions. It must be remembered that in this test air is excluded and water is present in excess so that certain important factors are excluded from affecting the reaction.

"Sandy" and "gravelly sandy soils" all gave smaller volumes of hydrogen than the "silt loams" and "clays," and the evolution of gas appears to depend directly upon the fineness of the soil. It may be found that the comparison of corrosivity of soils by the hydrogen-evolution method should only be made with soils having the same degree of fineness or texture.

In the case of two soils investigations were made to see how closely the volume of hydrogen liberated corresponded with the weight of iron oxidised. In the case of Susquehanna clay agreement was very close, while with Denver sandy loam somewhat more iron had been oxidised than was indicated by the hydrogen released. The estimation was performed by dissolving in hydrochloric acid and measuring the hydrogen liberated.

Particulars are given of a method of estimating the amount of non-corroded iron by means of mercuric-chloride.

For continuously observing the corrosion of iron in soil a method involving the measurement of magnetic flux is under investigation. By this method pulverised iron and soil mixtures in specimen tubes are placed in a solenoid coil and tests are made using a ballistic galvanometer to determine the magnetic flux in the specimen with a definite current in the solenoid. The flux will depend upon the amount of iron present in the uncorroded state and tests may be made upon any given specimen at any time without interrupting the reaction, and therefore continuous records can be obtained over any period of time. The conditions in this test as regards presence of moisture and air may be made to simulate field conditions.

The chief difficulty with the magnetic method is the production of uniform mixtures of soil, iron and moisture with a definite degree of compactness.

It is emphasised that the work done up to the present has been limited to developing a laboratory technique and that there is still considerable work to be done before the work can be standardised so as to permit comparison of the results of different workers. E. R. B. S.

364. Methods of Shutting-off Bottom Water. H. B. Hill and Chase E. Sutton. *The Oil and Gas Journal*, July, 1928, **26** (49), 90, 92, 180, 182, 183.

The Wortham Field, Texas, started to show water after a comparatively short period, and the position became so serious that the Bureau of Mines undertook a detailed engineering study of the field. The benefits of bottom-hole plugging in the Powell Field suggested a similar course at Wortham, which started in February, 1925, and had good results. As indicating the success of these operations it is estimated that 86 successful wells showed an increase of 11,400 bbls. of oil per well, and 35 unsuccessful wells showed a loss of 2,938 bbls. per well, giving a daily increase in production of 5,656 bbls. Water production showed a decrease of 33,500 bbls. per day.

These methods of bottom plugging are in common use, tubing with pressure, dump bailer and lead wool.

In some cases plugging off water in one well increased the production of oils in neighbouring wells.

A well should be plugged as soon as possible after the appearance of large quantities of water and the success of a job should be judged by its effect in the water production.

Before plugging is attempted, the following data is desirable :—(1) Junk present in the hole, (2) amount of perforated pipe in the hole, (3) size of perforations, which should be $\frac{3}{4}$ in. or larger, (4) condition of the water string, a leak in which may be the water source, and (5) the well's drilling and production history.

Any good oil-well cement can be used, and should be mixed with water not in excess of 45 lbs. per sack, the final mixture being as thick as the pumps can handle. Important steps in cementing are :—(1) Arrangement of surface equipment, (2) examination of tubing, (3) determination of exact depth of well, (4) cut a 4-ft. pole $\frac{1}{2}$ in. in diameter less than inside diameter of tubing and nail a snug-fitting washer on the end, (5) pack off tubing ring and tighten set screws in casing head, (6) tie one end of rotary hose to the standpipe and other end to the tubing or the elevators, (7) tighten bull ropes, (8) pack off tubing with wet cement sacks below spider, (9) use a short joint of tubing at top of string, (10) remove plug from $2\frac{1}{2}$ in. T at top of tubing and introduce wooden plug, leaving the $2\frac{1}{2}$-in. iron plug off until all water is pumped ahead of the cement, and (11) screw the $2\frac{1}{2}$-in. iron plug back into the T when the cement reaches the top of the tubing. Raise tubing 1 ft. from bottom and pump cement on to wooden plug, which acts chiefly as an indicator to show when the cement reaches bottom. When the plug reaches the bottom the pump will stop and should then be shut off, the tubing being raised high enough to allow the plug to pass out at the bottom. As soon as the cement reaches bottom the tubing is raised 4 ft. higher than the top of the desired plug, packed off with wet cement sacks at the casing head and the relief gate closed. After maintaining pressure of about 400 lbs. for three minutes the relief gate is opened enough to give free circulation and maintain a pressure of about 250 lbs. for at least six hours. The tubing should then be pulled in the clear and the well left untouched for seventy-two hours, when the tubing is lowered and the top of the plug located.

In a case where it was not possible to obtain returns, a plug was successfully built up as follows. The well was rigged as usual, but the cement box was placed so that the cement could be dumped into the tubing after the wooden plug had been pushed down. A swivel was used in place of the T and iron plug. As soon as the plug hit bottom the tubing was raised in the clear without packing off or applying pressure.

The main conclusions drawn from the study of the Wortham Field are :—

1. Wells completed immediately above the water contact have the highest initial production and show water at an early date. Wells completed some distance above the water contact have smaller initial production, but are not menaced by water for a longer period.
2. Rapid drilling and consequent large flush production resulting in sale of products in a falling market, may reduce profits.
3. Closer spacing of wells results in higher percentage recovery.
4. If coring had been done the results of plugging would have been improved.
5. Generally, bottom-hole plugging has been successful.
6. More care should be used in obtaining formation and production records.
7. Plugging requires careful analysis before work is started.
8. The sooner a well is repaired after water appears the greater the benefits.
9. No well should be considered not repairable until reasonable evidence of failure is obtained. G. S.

365. EXPERIMENTING ON CRUDE RECOVERY. J. L. Dwyer. *Oil and Gas Jnl.*, 1928, **26**, (49) 38, 154.

The Oil Recovery Section of the U.S. Bureau of Mines Experimental Station at Bartlesville, Okla, are conducting experiments regarding important problems in oil recovery work. The first problem is the determination of the quantity of oil originally present in a sand and the percentage recovered by ordinary methods. The second is the relation between depletion by ordinary methods and by repressuring methods. Thirdly, the relative efficiencies of compressed air and natural gas in repressuring operations and, fourthly, the formation of bad oil and inert incombustible gas in repressuring and the prevention of these troubles.

The laboratory is equipped with a unique high-pressure gas storage system, capable of holding 2000 lb pressure, and consisting of 100 high-pressure helium gas cylinders connected to a pressure header system. The duplication of underground conditions was recognised to be impossible and the comparative method was adopted, all variables other than that being studied being eliminated as far as possible. The materials at present being used are the Wilcox sand from Roff, Okla, Bartlesville crude of 33° A.P.I. gravity and Bartlesville city natural gas.

Two 6-in. flow tubes, 6 ft. long, equipped with miniature oil wells and flanged ends are tamped tightly with dry Wilcox sand and the heads replaced. The tubes are purged with three charges of gas at 100 lb pressure to remove all air and are then charged with gas at a pressure equal to that to be used in the experiment. The gas is then displaced by oil, and when full is connected as part of a circulating system in which the oil is pumped by high-pressure pumps. After about one hour's circulation the tubes are placed in a constant temperature water bath at 70° F., and circulation continued until the gas factor is constant throughout. After determining how much oil can be recovered by the expulsive force of the dissolved gas at various initial pressures, the tubes are repressured with air or gas to determine the recovery from the depleted sands by various methods.

It has been noted that compressed air introduced into oil sands is changed to a carbon dioxide and nitrogen mixture, and that casinghead gas soon becomes inert and useless. The character of the oil is also badly affected. G. S.

366. Landing and Cementing Long Water Strings in California. J. B. Case. *Oil and Gas Jnl.*, 1928, **26** (49), 74, 96, 101, 112, 124, 127.

The fundamental principles of the landing and cementing of a water string are, (1) setting the shoe in a suitable formation for impervious bonding, (2) the cement to be behind and completely around the casing, (3) the establishment of equilibrium pressure in the hole, and (4) the allowance of sufficient time for the cement to harden and cure. The most favourable conditions require sufficient space between the walls and the casing to insure a continuous wall of cement around the casing, and when circulating mud fluid or cementing a greater velocity of the ascending fluid outside the casing than of the descending column in the casing. The maximum size of hole for a given size of casing is determined by the formula $D^2 = 2d^2$ where D = diameter of the hole and d = inside diameter of the casing. The drilling of the hole should be carried out so as to obtain the best cementing conditions, and the hole should be kept as straight as possible and kept truly cylindrical. Where the exact cementing point is located by coring, a pilot hole is drilled ahead with the core barrel. It is important that the pilot hole should be centred. If the pilot hole is drilled a considerable distance below the oil sand it must be plugged before the water string is cemented.

Before handling casing, all mechanical equipment should be subjected to special inspection and a definite plan of procedure should be formulated. The more important points in procedure are (1) whether the casing is to be floated into position or not, (2) the determination of the exact depth of the shoe when cemented, (3) the method by which the cement is to be placed in the hole, (4) whether the casing is to be moved to pass collars during the displacing of the cement, (5) the position of the shoe when the cement starts out of the casing, and (6) how much casing is to be run in before establishing circulation. Care should be exercised in handling casing, and all defective joints should be discarded when making up a string. It is preferable to remove the rotary table when running casing and to support the spider on special timbers, and careful check should be kept of the number of joints lowered and their length when screwed up. The number of lines used in handling casing depends on its weight and the friction on it, a general average being 6 for 3000 ft. weighing up to 50 tons, and 8 for over 3000 ft. from 50 to possibly 80 tons.

Two principal methods are used for cementing, in one the cement being separated from the mud fluid ahead and behind by barriers, and in the other no barriers being used. The cement mix is 78 cu. ft. of water to 100 sacks of cement, making 125 cu. ft. of cement slurry. G. S.

367. Temperature in World's Deepest Wells. C. E. Van Orstrand. *Oil and Gas J.*, 1928, **48**, 39.

A brief history and description of the five deepest oil wells in the world to-day, namely, Goff (7,386 ft.), Lake (7,579 ft.), Wiley-Athens No. 6 (7,591 ft.), Ligonier (7,756 ft.) and Olinda (8,201 ft.).

Temperature-depth tables are given and computations for depths up to 10,000 ft. Conclusions reached are that the above remarkable depths are only milestones along the pathway of progress and future production may be expected from much greater depths. H. S. G.

368. Development of Hard Facings in Oil Drilling Equipment. W. F. Stoody. *Oil and Gas J.*, 1928, **48**, 39.

A brief review of the development of the percussion and rotary drilling bits and a description of the new type bit with hard facings.

Modern bits have additional cutting surfaces on the circumferential edge that will remove material there in the same ratio that the part of the bit nearer the centre removes its allotted formation. Hard facing at first consisted of laying the bit down upon a copper form and welding it out to size by depositing with the electric arc a bead of oil tempered spring steel, finally quenching whilst red hot with water. Later experiments were tried of building up the bit to original size with high carbon rod and not chilling or tempering in order to leave the bit and added material as tough as possible.

Next a high carbon rod was coated with a mixture of tungsten, manganese, chromium and carbon and applied in a bead to the face or cutting edge of the bit but deposition was too uneven. To overcome this alloys were placed inside a sheath and the sheath rolled and formed into a tube, but the demand was for something still harder. The next type of self-hardening rod placed on the market was a cast rod consisting chiefly of alloys.

The requirements of a good hard-facing rod are: (1) It should be applicable either by the electric or oxy-acetylene process. It should perfectly amalgamate or weld to the parent metal. (2) It should have a Rochwell hardness of 56-C. to 58-C. and still retain a cohesiveness of 100. (4) Its hardness should be inherent. (5) It should retain its hardness at red heat.

In hard facing the tool is made up of two metals, one having a low and the other a high degree of hardness, the hard material on the face of the tool is placed in the direction of rotation and does not wear off so rapidly as the softer metal behind it. Were the two materials nearly the same hardness there would be practically no undercut or clearance.

In order to fill the need for one tool for all formations Borium metal was developed and is the hardest material known except the diamond. Borium fragments are welded to the bit with a high carbon or mild steel rod, then covered up with stoodite or other hard-facing rod, the oxy-acetylene process being used. A Borium mounted bit costs about four times as much as a hardfaced bit but drills about five times more distance. H. S. G.

369. Breaking Petroleum Emulsions. M. de Groote and W. C. Adams. U.S.P. 1,656,622, Jan. 17, 1928.

A process for breaking petroleum emulsions by subjecting them to the action of a treating agent formed from tar acid oil, a sulphonic body and a soap body combined in such a way that the non-hydroxy hydrocarbons of the tar acid oil act simply as a diluent. The treating agent may be used in the form of an ester, or soluble or insoluble salt. H. S. G.

370. Breaking Petroleum Emulsions. M. de Groote and W. C. Adams. U.S.P .1,656,623, Jan. 17, 1928.

The emulsion is subjected to the action of a treating agent consisting of a mixture comprising a condensation product containing a sulphonated hydroxy poly-cyclic aromatic chemically combined with a soap-forming radical. In a preferred example oleic acid is mixed in molecular proportions with beta naphthol and the mixture sulphonated by means of excess of sulphuric acid. The product is washed and then converted into an ester or water soluble salt. H. S. G.

Petroleum Geology.

371. GEOLOGICAL FEATURES OF SUNSET FIELD. H. R. Farnsworth. *Oil and Gas J.*, **26**, **47**, p. 72.

The "Sunset" oil field embraces that portion of the oil-producing district in Kern County, California, situated along the south edge of Medway Valley and in the Temblor foothills, in the vicinity north-east and south of the town of Maricopa.

The geological and structural history of this field has been adequately covered by Arnold, Johnson and Pack (*U.S. Geol. Sur. Bull.*, No. 406, 1910, and *U.S. Geol. Sur.*, Prof. Paper, No. 116, Part 1, 1920) and their conclusions are generally accepted as correct

The coast ranges of California form an "Anticlinorium," in which the rocks, mainly sedimentary, Jurassic to post-Tertiary, are greatly folded and faulted. This mountainous belt is composed of several ranges which, though approximately parallel to each other, trend somewhat more easterly than the belt as a whole, cutting obliquely across it.

The "Temblor" range is the easternmost of these ranges and is essentially a great anticline broken by faults along its south-west flank. It is an assemblage of small anticlines and synclines, the combined effect of which represents the general uplifting of the area. The central portion of this range exhibits rocks older than those exposed in the foothills to the north-east, and were subjected to folding in the late middle Miocene period before the younger Miocene formations were deposited.

The south end of the Temblor range was, and is, composed of diatomaceous (Maricopa) shales, with lenses of coarse material.

The north-east flank of this Maricopa shale had assumed the form of a monocline, dipping to the north-east; its strike closely parallel to the main axis of the range, bearing 46° W.

Subsequent to this folding (from upper Miocene age) a long period of deposition ensued. The beds now containing the oil, being composed of detritus, brought down into the San Joaquin valley from the high land situated where Temblor and San Emiglio mountains now stand, most of the debris probably coming easterly from the Temblor range area. The lower of these detrital beds (McKittrick group) of upper Miocene and perhaps Pliocene age was composed of poorly consolidated sands, clays and gravels—deposited in a sinking basin in marine and estuarine waters at first occupying only the San Joaquin Valley, but later encroaching upon and submerging the high land that then stood on the present Temblor range site. The upper McKittrick beds (Pliocene and Pleistocene) were deposited in brackish or fresh-water lakes and extended south-westerly up the slope of the high land and beyond the north-easterly regions of lower relief, then occupied by the Etchegoin. The lower McKittrick group of beds rest unconformably on the monoclinal surface of the Maricopa shale. These horizontal beds were deposited with overlapping.

The general approximate bearing of N. 46° W. is an important function of the extant monoclinal form of the surface of the underlying Maricopa shale, which closely parallels the main axis of the Temblor range.

It results that, having given the location of a point on the shore or contact line of any bed of the lower McKittrick group, the approximate position of the remainder of the short line of that bed along the north-east flank of the southern portion of the Temblor range may be delineated by simply drawing a line on a bearing of N. 46° W. and S. 46° E. through the given point.

The author explains with the aid of plates the use he made of the above in estimating the position of the top of the "Top Oil" zone with reference

to the surface of the Maricopa shale, and of estimating the position of the shore lines of individual oil-bearing sands.

Subsequent to the deposition of the McKittrick beds a fresh set of flowing movements gave rise to secondary anticlines, which are only approximately parallel to the course of the main range, trending more to the east; the difference in bearing between the course of these secondary folds and the trend of the range increases towards the north-east.

The accumulation of oil seems to be largely governed by these obliquely-trending anticlines, as it is above these folds that most of the oil is found. They increase in size and number southward along the western border of the San Joaquin Valley until at the south end of the Temblor range they develop into a complicated system.

The "Thirty-Five Anticline" is one of the chief structural features in the foothills of the main Temblor range west of Midway Valley and south of the "Globe" anticline.

It trends south-east and edges out into the outermost foothills, finally plunging beneath Midway Valley. On the south this anticline is separated from the Temblor range by the "Phœnix" syncline. At the south end of the Temblor range the bearings of the structure lines exhibit a marked change from a south-east to an east trend—this is due to the eastward trending San Emiglio Mountains. The structure, typical of the easterly margin of the coast range, is thus gradually transformed into that of the Sierra Nevada—the Midway Valley being divided into two different structural zones.

The article concludes with the mention of various wells by the Union Oil Co. and the Standard Oil Co. ("Tar Sand") and points out that with the foregoing geologic and structural features as a basis an appropriate application of the known stratigraphy of the field for the purpose of estimating the productive capacity of any property situated therein will prove to be convincingly accurate. J. E. M. H.

372. The Causes of the Premature Decline of the Oil-Bearing Strata of Hanover. D. Edmund Laske, Hanover. *Petr. Zeit.*, April 1, 1928, p. 426.

The writer, for 20 years past, has held the view that the main oil deposits are not to be found on the borders of the Salzgebiege, but are remote from there. Latest results afford proof of this.

The richest fields at Nieuhagen, Forstort Brandt, Hänigsen and Oberg, have proved unproductive from most wells, but Mr. Laske is convinced that a more profitable result can be obtained.

By this premature filling in of all boreholes after exhaustion from the first oil-bed horizon, the enormous cost of the contract boring was raised out of all proportion to the oil-production, so as to become a non-payable concern.

It is clear that oil horizons at greater depth could be reached far more cheaply in these boreholes that had reached the first horizon than by new ones now being carried forward.

Also, far too many useless experiments have been tried, increasing the cost of boring. Despite numerous failures, new boreholes with different apparatus were made which met with oil only too seldom, but mostly added to the number of dry holes.

A more systematic method of boring technique is required, since the opposing counsels of skilled borers and geologists have only resulted in failure. Just as only a skilled mining engineer can carry out mining operations, so these boring operations must be carried out by boring experts and not by typical geologists. In view of the facts, this filling-in of the boreholes reaching the upper oil horizon was a mistake.

Again, the boreholes are too far apart, some not reaching oil and not fully utilized. They clearly reach different horizons moreover. The decrease in yield of oil in the Hanover oil field may thus be accounted for as due to a scheme of boring that has not been logically and systematically devised and carried through.

Sometimes by cracks and crevices the boreholes tapped great quantities of oil forced up by high pressure of gas—at other times, in the oil sands, the pressure was so slight that the oil did not flow out freely, but should have been pumped. When the oil flow diminished from failure of gas pressure, it was considered as exhausted and boring was suspended.

It can be inferred that all the oil from sand and sandstone belonged to the upper horizon, but that the rest came from lower horizons.

The shallow boreholes to the first horizons must be carried deeper to the middle Jura (Dogger) and then black Jura (Lias), which may safely be considered as oil-bearing. Intermediate boreholes are recommended also in the richer oil zones, where the spacing has been too great. J. E. M. H.

373. Geologic Factors in Water Flooding. Paul W. Torrey. *Oil and Gas J.*, May 17, 1928.

From a fundamental standpoint, the character of the reservoir rock is the deciding factor in the success or failure of water floods.

The selection of properties is probably the most important feature of flooding operations.

Geologic Factors Influencing Recovery by Flooding.

(1) Effects of configurations in the top and bottom of the oil sands on the rate and direction of flood movement :—

(*a*) Major Structural Features.—A low dip is favourable, and where the dip is over 1°, flooding up dip is the logical procedure.

(*b*) The Strike Theory.—The flood appears to move fastest in the direction of the structural strike of the sands, especially such as lie relatively flat.

(*c*) Sand Thickness.—Where irregularities occur in the bottom of the sand it is most advisable to flood from thin to thick sand. Where the sand possesses a fairly homogeneous lithology and oil saturation the wells located on the high spots of the sand are usually the best producers, wells located on low spots giving very poor results.

(2) Porosity.—It would seem that the actual porosity of a sand, within certain limits, has a very slight effect upon flood movement.

Permeability.—Sands possessing a fairly high porosity and a low permeability seem best adapted to water flooding.

(3) Grain Size.—Fine grained sands possess a distinct advantage over coarse grained for flooding purposes, since they more effectively resist depletion of their oil-content by natural methods of production owing to their lower permeability and their larger area of grain surface. The saturation of the sand is a most important factor in determining the amount of oil that may be recovered by water flooding. Wider spacing in the wells causes more oil to be left easily recoverable by the water flood under uniform sand conditions.

(4) Sand Cement.—Sand grains coated with silica are better adapted for flooding purposes than those coated by lime or iron oxide.

(5) Effect of Oil Gravity and Natural Rock Pressure.—High gravity oil is better adapted to flooding than low gravity oil.

Natural rock pressure, above 5 or 10 lbs., is not especially helpful in effectively flooding an oil sand, but even a small back pressure will seriously affect production. J. E. M. H.

373A. Evolution of the Eötvös Torsion Balance. W. H. Fordham. *Oil News*, 1928, xxiii. (807), 469–470.

This article is supplementary to a previous communication by the same author ("Oil Finding by Geophysical Methods," *Journ. Inst. Petr. Techn.*, 1925, **11** (52), 448–470), since the publication of which a number of fundamental changes have been made in the design of Eötvös balances.

These changes have been embodied in a new instrument, which will revolutionise gravity surveying, and a brief account of the considerations which led to its design, and a description of the instrument itself, follow.

Reference to equations (1), (6) and (12) of the previous article and to the two patent specifications mentioned later will confirm the soundness of the conclusions reached.

Attention is called to two points in connection with the balance formula and the Terrain Correction formulæ :—

(*a*) The normal type of Eötvös balance measures four quantities, two giving the gradient of gravity and two the curvature values. In gravity surveying for minerals the gradient has the greater practical value, the curvature value being completely neglected in many cases. Thus an instrument measuring gradients only would be nearly as useful as the normal balance.

(*b*) The incidence of terrain effects depends on the height above ground level of the centre of gravity of the weights on the suspended beam system, and this height should be as large as possible.

It is also of practical importance that (*c*) the instrument be so protected that it can be moved in wet weather without risk of damage, and (*d*) from the point of view of surveying costs, should be easily transportable and should allow of observations being made by day.

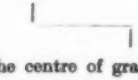
The early visual balance was designed to measure gradients and curvatures with equal accuracy, and was large and unwieldy. Nearly two hours were needed for the beams to come to rest, and readings could only be obtained at night, with the result that only one station could be completed in 24 hours. The centre of gravity of the weights was about 70 cm. above ground.

The introduction of the automatic instrument reduced the labour of taking readings. The placing of the optical system inside the balance case made a more compact and easily protected instrument, and with improvements in the air damping and designing a more efficient protecting hut, readings could be taken every hour. With these improved instruments two stations could be completed in 24 hours.

While these changes were taking place a number of smaller instruments were introduced. Used on heavy tripod stands, the centre of gravity of the weights is kept high, but while reduced dimensions gave greater portability, the instruments were less sensitive and have not been very satisfactory. Three stations can, however, be completed in 24 hours.

All the balances are of the Eötvös type, with straight horizontal beams, to one end of which a weight is fixed, a second weight being suspended from the other end by a long thin wire. It is not clear that such instruments have given good results by day.

The first satisfactory departure from this type of beam was made by Prof. Schweydar in his "Z" beam balance. The beam is stiff bar, shaped thus :



with weights at the end, the centre of gravity being about level with the horizontal part of the beam.

The "Z" beam instrument, a double beam automatic balance, is smaller than the old full-sized instruments, is comparatively compact and has a high centre

of gravity. Day readings are obtainable and three or four stations can be completed in 24 hours.

The first instrument for gradients only was that of the North American Exploration Co., which had two inclined beams, straight bars with weights at the ends, at right angles to each other instead of parallel. Although the instrument is influenced by curvature values it does not measure them and no attempt seems to have been made to render it more sensitive to gradients than to curvatures. Only three settings are needed at each station and a larger number of stations can be completed in a given time than with the ordinary balance, which needs four settings.

In B.P. No. 272,511 of 1927, Prof. Kogbetliantz proposed an instrument influenced by gradients only. The beam has three equal arms 120° apart, to the end of one of which a weight is fixed, other weights of equal size being hung on wires of different lengths from the ends of the other two arms. An instrument with a single beam requires four settings, and a three-beam instrument was therefore proposed to reduce the number of settings, but apparently the mechanical difficulties have not yet been overcome.

The general solution to the "Gradiometer" equation was obtained by Messrs. Shaw and Lancaster-Jones at about the same time that Prof. Kogbetliantz found his special solution. In B.P. 275,386 of 1927 they pointed out that a beam carrying three or more weights at two differing levels is influenced by gradients only under certain conditions.

As the beam system is influenced by gradients only, the form of the instrument constants in the gradient terms of the balance equation alone has to be taken into account in the design of the beam, thus heavier weights and shorter arms than in the Eötvös type can be adopted with advantage, the difference in level between the upper and lower weights being kept as large as possible.

An instrument designed on these lines, the "Gradiometer," is now on the market. It is a single-beam visual reading instrument, the beam being a circular ring, on which are fixed two or more weights, and which carries a vertical standard with another weight at the end. A very sensitive ribbon section, torsion wire is used, enabling the beam dimensions to be comparatively small. The vibration period of the beam is about eight minutes, so with an efficient damping system the beam comes to rest in less than 25 minutes. The small size of the beam and also the "lower" weight being above the beam, enables a compact and easily protected design of instrument to be adopted. The centre of gravity of the weights being above the beam allows the height of this centre to be large in comparison with the height of the instrument. The balance proper, in a double-walled case, is totally enclosed in a cylindrical aluminum cover 8 ins. in diameter. The upper part of the instrument is about 10 ins. in diameter and 30 ins. high, and can be placed in a waterproof bag when moving on a wet day. The lower part is a tripod stand 18 ins. high, the balance, when assembled, standing just over 4 ft. and weighing 90 lbs. Observations can be made by day if a good shelter tent is provided. Four settings are required at each station, each setting requiring less than 25 minutes, so that a station can be completed in 2 to 2½ hours. From these points it will be seen how nearly the new instrument fulfils the ideal condition (*a*), (*b*), (*c*) and (*d*) at the beginning of this article. G. S.

Coal and Shale.

374. Improvements in Catalytic Gas Reactions. I. G. Farbenind. A.-G. E.P. 274,904, 1928.

The invention aims to secure the highest possible catalytic effect in catalytic gas reactions, such as in the synthesis of ammonia from its elements and the

production of methanol by the reduction of oxides of carbon, but particularly in the destructive hydrogenation of carbonaceous materials. To attain this end, the catalyst is brought into a state of suspension throughout the reaction space, the particles being whirled about by a gas which may or may not participate in the reaction. The catalyst is separated from the reaction gases before the latter reach the cooler parts of the apparatus. H. G. S.

375. Improvements in the Production of Hydrocarbons from Coal, Tars, Mineral Oils and the like. I. G. Farbenind. A.-G. E.P. 286,825, 1928.

Coal, tars, mineral oils and the like in a vaporised or finely-divided state are exposed to the action of an electric arc in the presence of about one-half or less of the quantity of water vapour required for the production of water gas, so that acetylene is produced in appreciable amount. The water vapour may be replaced by hydrogen, nitrogen, carbon dioxide, carbon monoxide or their mixtures and the original materials may be introduced into the electric arc in a heated condition. Example 1.—Hard-coal dust with 6 per cent. of tar-forming constituents is passed through an electric arc in association with gases of combustion containing water vapour, the relative proportions being such that the mixture contains 200 to 300 grams. of water vapour to one kilo. of coal. About 50 litres of acetylene are obtained from 1 kilo. of coal, the consumption of current being 1 kilowatt-hour per 40 litres of acetylene. Example 2.—Brown-coal tar (500 grams.) finely distributed in hydrogen (1 cubic metre) is passed through an electric arc. The product consists mainly of unsaturated hydrocarbons, in association with water and a little hydrocyanic acid, about 300 litres of acetylene being obtained per kilogram of tar. The current consumption is 1 kilowatt hour per 50 to 70 litres of acetylene. Example 3.—20 parts by weight of coal tar and 1 part of hydrogen at 350° C. are passed through an electric arc. The yields by weight per cent. are :—acetylene 25 to 30, benzine 5, lubricating oils 5, hydrogen and heavy hydrocarbons 5 to 10. H. G. S.

376. Improvements in the Destructive Hydrogenation of Carbonaceous Materials. I. G. Farbenind, A.-G. E.P. 272,538, 1928.

In the destructive hydrogenation of coal, tars, mineral oils, pitches, resins and the like, it is essential to maintain the concentration of hydrogen in the reducing gases, especially when the latter have to be returned repeatedly to the plant. The gaseous products, after removal of constituents liquid at ordinary temperatures, are scrubbed under pressure with benzine, preferably with the benzine produced in the process. H. G. S.

377. Improvements in the Manufacture and Production of Valuable Hydrocarbons or Derivatives thereof from Coal, Tars, Mineral Oils and the like. I. G. Farb. Ind. A.-G. E.P. (*a*) 258,272, 1928; (*b*) 285,923, 1928; (*c*) 286,678, 1928; (*d*) 286,679, 1928; (*e*) 286,680, 1928; (*f*) 286,681, 1928.

These processes are concerned with the "destructive hydrogenation" of coal, tars, mineral oils and similar carbonaceous materials by the action of hydrogen or gases containing hydrogen at elevated temperatures and at pressures exceeding 50 atm. in the presence of catalysts, the object being the production of valuable liquid products, chiefly of low boiling point. The reducing gases may be hydrogen, hydrogen mixed with nitrogen, water gas, hydrogen containing sulphuretted hydrogen, water vapour or hydrocarbons such as methane. Alternatively the hydrogen may be produced in the

reaction vessel, *e.g.*, by the interaction of water vapour and carbon monoxide or hydrocarbons. The catalyst specified are :—

(*a*) Compounds of silver or copper, alone or mixed with difficultly reducible metal oxides, or carbonates which form difficultly reducible oxides. Other additions to the catalyst may be made, *e.g.*, metals of the 8th group of the periodic system or their compounds.

(*b*) Difficultly reducible oxides, or carbonates forming such oxides of metals of the 3rd to the 5th group, together with metals of the 8th group, *e.g.*, iron, or their compounds.

(*c*) Oxide or carbonate of lithium, magnesium or zinc, together with metals of the 8th group or their compounds.

(*d*) Compounds of cadmium, lead, bismuth or tin, together with difficultly reducible oxides or carbonates forming such oxides and metals of the 8th group or their compounds.

(*e*) Oxide or carbonate of uranium, together with metals of the 8th group or their compounds.

(*f*) Oxide or carbonate of manganese and metals of the 8th group or their compounds.

An example given under (*b*) quotes the formation of a product containing upwards of 50 per cent. of petrol by continuously passing brown coal producer tar over a catalyst consisting of porous material coated with vanadium oxide, the pressure and temperature being 200 atm. and 500° C. respectively.

H. G. S.

378. Improvements in or relating to Low Temperature Carbonisation. Kohlenveredlung A. G. E.P. 267,505, 1928.

The invention has particular reference to the treatment of coal, in thin layers, in externally heated retorts, and consists in the admission of gas, *e.g.*, water gas or generator gas, into the retort. The added gas is preferably introduced under a slight positive pressure and may be advantageously preheated by the waste gases from the still or by the hot coke. Further, where means are provided for drawing off fractionated gaseous products of distillation, additional gas may be separately brought to the fractions.

H. G. S.

379. Method of Producing High-value Oils from Raw Coal by Low-temperature Distillation and Hydrogenation. R. Feige. E.P. 274,465, 1928.

Raw coal is subjected to low-temperature carbonisation. The solid residue is in part utilised to generate electric current which, in turn, is employed to decompose water into its elements. The oxygen produced is used to gasify the remainder of the coke, the gases from which, together with the hydrogen prepared from the water, are treated in a hydrogenating plant with, in certain circumstances, the addition of the distillation tar or gas or both. H. G. S.

380. Improvements in or relating to the Distillation of Solid Carbonaceous Materials. H. Nielsen and B. Laing. (*a*) E.P. 287,037, 1928 ; (*b*) E.P. 287,381, 1928 ; (*c*) E.P. 290,551, 1928.

(*a*) The process is concerned with the continuous distillation of coal, shale, torbanite, etc., by means of a counter-current of heated inert gas such as water gas, producer gas or combustion gases, the volume of the heating medium being such that the vapour tension of the volatile products is reduced

to cause them to be driven off at temperatures below that at which they normally volatilise. The temperature at which the material is treated in these conditions does not exceed approximately 450° to 550° C. If substances containing a large percentage of oil (*e.g.*, oil shales or torbanites) are employed, the volume of distilling material is regulated so that, after distillation is complete, it may be cooled to 80 or 100°C. without causing the heaviest or highest boiling point oil fraction to condense. Fusible or coking coals may be successfully dealt with if they are subjected to a preliminary heat treatment with gases containing an oxidising constituent or with a gas containing from 2 per cent. to 8 per cent. of free oxygen ; or, alternatively, a controlled volume of free oxygen may be added to the heating gases before or during distillation. The primary oils are claimed to contain a large percentage of high boiling fractions from which, after removal of tar-acids, kerosine and wax, lubricating oils possessing a static coefficient of friction from 0·1 to 0·185 at normal temperature may be extracted. In operating the process, the oils in suspension in the heating gases are withdrawn from the apparatus at different points along its length and additional volumes of heating or carrying gas may be admitted to the combined retort and heating gases. The solid residue is substantially free from deposited graphitic carbon, contains from 4 per cent. to 15 per cent. of volatile matter and is free-burning either in lump or pulverised form. In the example quoted, a bituminous coal, having a volatile content of 30 per cent. after preheating to 150°C., was distilled in a rotary retort in counter-current to a controlled volume of superheated water gas (55,000 cubic feet per ton of coal). The gas entered the retort at 750°C. Oil and a small volume of gas were evolved at 210°C., and at about 550°C. all the oil-giving materials were removed. The products recovered were 20 gallons of oils, 15 gallons of ammoniacal liquor and 2,500 cubic feet of stripped retort gas combined with water gas. The oil, after fractionation and refining, gave 3 per cent. of petroleum ether, 3 per cent. naphtha, 3 per cent. benzine, 40 per cent. kerosine, 30 per cent. lubricating oils and 4 per cent. paraffin wax. The fraction boiling over 300°C., *i.e.*, lubricating-oil base, after acid refining and filter pressing, gave rather dark-red oils having a marked green fluorescence, the yield of all grades of lubricants being 15 per cent. of the total original crude tar.

(*b*) The time factor and temperature gradient during distillation are so controlled that a temperature difference exceeding 150°C. is avoided between the temperatures of the centre and periphery of any piece of material. The raw coal is crushed to below 2-inch cube and fed continuously for a period of approximately two hours through progressively increasing temperature zones produced by heating gases having a heat gradient diminishing from about 650°C. down to 110°C. The volatile products are withdrawn from the plant at approximately the temperatures at which they are evolved so that re-evaporation or condensation is prevented.

(*c*) This invention claims the primary oil containing heavy lubricating bases prepared according to the previous specifications. The primary oil, or the neutral oil obtained by removing from it the acidic and basic constituents, contains over 9 per cent. by weight of hydrogen and has specific gravities ranging from 0·95 to 1·080 at 15°C. The lubricating oil itself possesses a static coefficient of friction of 0·1 to 0·185 at normal temperatures. H. G. S.

381. Improvements Relating to the Distillation of Coal and Similar Carbonaceous Substances. Low, Temp. Carb., Ltd., and C. H. Parker. E.P. 287,830. 1928.

Coal or other carbonaceous material is distilled at low or medium temperatures in two stages, firstly in a retort which is heated either externally, or

internally by means of hot gases, and secondly in a retort or chamber to which heat is not applied. The junction between the upper and lower retorts is closed by means of a door and the volatile products are withdrawn from the upper ends of each retort, the gas ascension pipe from the bottom retort entering the gas outlet from the upper retort at a point to the rear of an anti-dip valve.

H. G. S.

382. Improvements Relating to the Distillation of Carbonaceous or Bituminous Substances. Kohlenveredlung A. G. E.P. *a* 286,404. 1928. *b* 287,627. 1928.

The inventions relate to the carbonisation of bituminous materials in the form of powder or fine grains by suspension in heated gases.

a.—Hot generator or water gas, which is produced from the solid residue obtained in the process, is employed as the carrier or heating medium and is fed immediately after leaving the gas producer, together with the raw material, into a distilling chamber. The latter may consist of a number of horizontal or inclined tubes which may be also externally heated, or they may consist of a series of vertical concentric tubes, the emulsion of gas and powdered material entering at the base of the inner cylinder and the products leaving the bottom of the outer vessel. It is preferable to carry out the distillation in stages. To this end, the coal and heating gases travel generally in counter current through a plant built up of a number of retorts, but through each retort, the raw material and heating medium travel in the same direction, the solid and gaseous products being separated between the successive stages.

b.—The carrying or heating gas is maintained at a temperature exceeding 800°C., and the contact with powdered coal is regulated for such a short time that the substance is subjected merely to low temperature carbonisation.

H. G. S.

383. An Improved Process and Apparatus for the Manufacture from Bituminous Shales of a Product Adapted for Dry Distillation. Patent-a-b Grondal-Ramen. E.P. 278,378, 1928.

The process is designed for the pre-treatment of bituminous shales which stick together during distillation. The shale is first crushed to a maximum size of 10 mm., thoroughly mixed and then made into balls with water in a rotating drum. The feed to the drum is so arranged that the material may be delivered to any desired part of the drum. The smaller balls are withdrawn and reintroduced for further treatment while the larger fragments pass through a drier.

H. G. S.

384. Distillation and Gasification of Solid Carbonaceous Material. Alfred Knight Croad (from Zieley Processes Corpn., Maine). E.P. 287,999, Feb. 14, 1927.

Coal and other solid hydrocarbon fuels are distilled and gasified at relatively high temperatures, the material being previously coated or impregnated with sodium chloride to break up its constituents when subjected to a temperature of 2500° F. and to enter catalytically into the reaction. Air and steam may be introduced into the reaction chamber and fresh quantities of sodium chloride may be added to expedite the reaction.

G. S.

385. Process for Distilling and Liquefying Coal. F. Bergius. U.S.P. 1,669,439, 1928. Appl., Nov. 24, 1926.

Coal is carbonised in gas retorts or coke ovens to produce ammonia, tar, coke and gas, the latter being divided into two fractions: (*a*) poor in hydrogen but of high heating value, (*b*) rich in hydrogen but of poor heating value. Gas (*a*), formed in the early stages of carbonisation, is utilised as town's gas. Gas (*b*), together with hydrogen obtained by gasification of the coke, is employed in the berginisation of a further supply of raw coal, which is made into a paste with the tar produced in the initial carbonisation. The residual gases of berginisation are sold as town's gas. H. G. S.

Abstracts.

Arranged by H. G. Shatwell, Ph.D.

Abstractors.—Miss W. S. Clarke, B.Sc., S. W. Cooke, B.Sc., P. Docksey, B.A., H. S. Garlick, B.Sc., A.I.C., W. N. Hoyte, B.A., R. Pitkethly, G. Sell, H. G. Shatwell, M.Sc.(Tech.), Ph.D., E. R. B. Skipwith, B.A., A. R. Stark, B.Sc., A.I.C., H. E. Tester, B.Sc.

General.

386. The Petroleum Industry of Venezuela. *Petr. Times*, 1928, **19**, 1047—1048.

This report, written by the United States Consul, describes the great development of the petroleum resources of the Maracaibo Basin in Venezeula during 1927. Pools promising commercial value were discovered in the district of Mara, State of Zulia, and in the central area of the district of Buchivacoa, State of Falcon. On account of over-production, in April, 1927, the number of rigs each company was to operate was reduced and locations to be drilled were restricted, but activities were concentrated on the La Rosa and Lagunillas fields. The excess production was met to a great extent by an augmentation of the tanker fleets of the main operating companies.

The production of oil in the Maracaibo Basin in 1927 totalled 62,817,088 barrels, *i.e.*, greater than 70% in advance of the corresponding figure for the preceding year. The production from the Lagunillas field during 1927 was especially important as it affected operations in practically every other field in the basin except El Mene and Aro and Tarra. In the early summer of 1927 the English company closed down its La Paz and Concepcion fields and so ceased operations in Ambrosio and Punta Benitez areas, and concentrated its efforts on the La Rosa and Lagunillas fields.

The exports of crude oil from the Maracaibo Basin in 1927 increased considerably over those of the two preceding years. The largest monthly exportation in the history of the basin was attained in December 1927, when the shipments amounted to 6,196,989 barrels. Also the gradual increase in the shipments during the second half of the year is attributed to a gradual increase of the tanker fleets on L. Maracaibo. W. S. C.

387. The Mineral Oil Industry in Italy. G. Weiss. *Petr. Zeitschr*, 1928, **24** (600—603).

A short historical survey of the oil and gas occurrences in Italy during the nineteenth century is first given in this article. The most important zone for the Italian petroleum industry to-day is the region of Emilia (S. Lombardy), where there are three oil-wells. The producing fields appear to be present in the mountainous regions, and the richest petroleum occurrences are found to be at the foot of the mountains.

Recently, during the years 1925—1927, the formation and development of the Agip petroleum company has resulted in the carrying out of several

geological investigations, chiefly by the application of geophysical methods. In March, 1927, borings were commenced at Abbateggio (S. Ancona) and at San Colombano al Lambro. Research on asphalt occurrences at Ragusa (Sicily) and on the petroleum occurrences in the Northern range of the Apennines was also undertaken. The region on the N.E. slope of the Apennines between Alessandri and Rimini, and the district of Emilia promise considerable production of petroleum in the near future.

The geological regions in which petroleum has been discovered in Sicily are on the eastern half of the island. Numerous gas occurrences and bituminous schists were well-known in W. Sirgenti prior to the Great War, and gypsum marls were found at Nicosia. The first boring undertaken by the Agip in Sicily was at Bivona. The volcanic soil of Italy and Sicily suggests the possibilities of rich petroleum occurrences. W. S. C.

388. Clays and their Application in Refining. G. W. Cupit, Jr. *The Refiner*. 1928, **4**, 69.

The vital factors in the application of clay to oil are the proper selection of the clay and its preparation. It is only recently that the application of "fine" clays to oils for decoloration and neutralisation has been of commercial value. The clay most generally used is Fuller's Earth. The clay is dug from the quarry and placed in storage bins from which it is transferred by belt conveyors to the crusher. The broken clay passes to a drier through which it passes countercurrent with the heat to a cooling tank. The dried material is then ground and sieved, the grades produced being 15–30, 30–60, and 60–100 mesh. The "fine" clay is mostly produced during grinding and approximates 15%.

The clay is tested by taking an average sample of about 400 grams and drying it in an oven maintained at about 190° F. The loss in weight is determined and the colour and fracture of the clay noted. The clay is then ground and sifted through 30 and 60 mesh sieves. The weights of the fractions coarser than 30, 30 to 60, and finer than 60 are determined and also the percentage of 30–60 finer than 60. The volatile matter of the 30–60 fraction is determined by heating 1—1½ grams to a bright red heat for 10 mins. The volume-weight is next determined by carefully filling a 100 c.c. cylinder. The weight in grams per 100 c.c. clay multiplied by 0·624 gives the weight in pounds per cubic foot.

To determine the filter yields, three percolators are charged with 65 c.c. of the burned 30–60 clay. The same cylinder used in the determination of volume-weight should be used. After filling, suction is applied to the bottom of the percolator for about 30 seconds to remove dust. The oil is then poured into the centre of the clay at the top and allowed to soak downward. When the clay has taken up all the oil, sufficient is added above the clay level to cause the oil to run into a weighed container at the bottom of the percolator. After the filter has started to flow, the oil level must not be allowed to fall below the clay level, otherwise an air pocket will be formed which interferes with the flow of oil through the filter. For spindle and neutral oils, the best filtering temperature has been found to be 90–120° F. and for bright stocks 180–200° F. The time of charging the stock, the time of the first drop into the receiver, and the time when the colour of the total filtrate reaches that of the standard sample are all noted. The yield in grams per 65 c.c. being known, the yield in barrels per ton of clay can be calculated.

In general practice, two-grades of clay are prepared, *namely*, H.B. [highly burned] clay and M.B. [medium burned] clay. The former contains approximately 2—3% volatile matter and the latter 7—9%. The former is used

for filtering cylinder oils and the latter for spindle, neutral oils and petrolatums. The oil is run through the filters under a pressure of 8–10 pounds.

In general three types of methods of regeneration have been applied, *namely*, (1) solvent method, (2) chemical method, (3) physical method. In the solvent method, both straight and mixed solvents have been used. Where this method is used it is found necessary to combine it with the physical method to obtain the results desired. It may also be used in conjunction with the chemical method.

After the clay has been well washed with naphtha, it is steamed for approximately 15 hours. The amount of clay treated at one time ranges from 40—45 tons. In starting a treatment approximately 6,500 gallons of sour water are pumped into an agitator and agitation begun with air and steam. The clay to be regenerated is then run into the agitator, after which 7,500 gallons of 30° Bé. sulphuric acid are added. The contents are then brought to the boiling point and held there whilst agitation takes place. After blowing 8 hours the contents are allowed to settle 8 hours, after which they are reblown 6 hours. This procedure is followed until the clay has been agitated seven or eight times. When the clay, on testing, is found to be satisfactorily treated, it is washed by filling the agitator with water to within a foot of the top, agitating and allowing to settle. Sour water to the amount of 8,500 gallons is drawn off to tanks through cocks above the clay line, 7,000 gallons of this wash being used in the next treat. Washing is continued until the water becomes clear and practically free of acid. This requires about 4,000 gallons per ton of clay treated. When washed satisfactorily, the mass is washed with as much water as possible and while still agitating the bottom cock is opened and the mass let down to the drying tank, where the water is drained off. Superheated steam is then passed through the drier for 12 hours at 10 lbs. pressure, and then compressed air, heated with superheated steam, for 24 hours. Sufficient amount of clay to make room for the next batch is then drawn off.

Results seem to indicate that the volatile matter content has little, if any, influence upon the decolorising power of a clay, as long as it is not present as hygroscopic water. Although it is assumed that a burnt clay of low weight per cubic foot has a greater decolorising power than a heavier clay, there are numerous exceptions. The ratio between the decolorising power of various clays is not the same for different stocks. It has been found that a large proportion of free silicic acid present is disadvantageous and that a large proportion of calcium and magnesium silicates is advantageous in its decolorising power. The decrease in decolorising power of a clay by regeneration will be least if the used clay is thoroughly well washed with naphtha to remove soakage and the organic matter remaining is burned off as well as possible. The effect of acid treating a used clay is to decrease its decolorising power when the clay has been burned only two or three times and to increase it when it has been burned ten or eleven times.

Clays, besides being used for filtering, are used in fire and steam distillation, one commonly used being magnesite. Usually a pressed paraffin distillate is used for the work. Results show that the use of clay lessens the tendency of the oil to become discoloured, and lessens its tendency to emulsify. Further, the Conradson carbon test and the sulphur content were both found to be less when clay was used in the still. S. W. C.

389. Nature of Petroleum Filtering Earths. P. G. Nutting. *Oil and Gas J.*, June 28, 1928, 138.

The theory is advanced that the activity of filtering earths is due to the presence of open O- and Si- bonds, which act by attaching certain alkyl or

weakly basic radicals of hydrocarbons. The result may be mere adhesion but in many cases is a surface reaction in which a film of organic silicate is formed over the surface of the filter grains. For this reason, the filtering earths, which are combinations of bases with the weak silica and alumino-silicic acids, must have the basic radicals removed, leaving open bonds ready to attach to the radicals of the oil to be filtered. Consequently, the filter is chosen or prepared to have terminal hydroxyl radicals which are then removed by heating, leaving the open bonds essential for filtering. The author claims that support is given to his theory by the fact that a spent Fullers earth cannot be washed clean even by the most powerful solvents in which the reaction products (silico hydrocarbons) are insoluble. Further, these products cannot be simple hydrocarbons because burning to 800° C. in air simply carbonises the film. Many of the best filtering earths must be acid washed before being dried for use: this treatment converts the terminal —OK, —ONa or —O_2Mg radicals into —OH radicals and salts, the latter being washed out and the residual product dried to form open bonds necessary for filtration. H. G. S.

390. The Oxidation of Hydrocarbons as a Technical Problem. F. Hebler. *Erdol und Teer*, 1928, **10**, 333–4.

The development of oxidation processes is described. Schaal (1884) oxidised petroleum and similar hydrocarbons in presence of alkalis, calcium chloride and alkaline carbonates by heating to 200°-300°C. in a stream of air or oxygen. Copper salts were also used as catalysts. Certain French processes in 1912 made use of sodium peroxide and steam, or ozonides or ozone in the presence of acids or alkalis. Grey (1915) effected oxidation by means of ultraviolet light in presence or absence of catalysts. Mathesius (1916) utilised nickel oxide or mercuric oxide and found that in a current of air at 103 to 120°C., 80 per cent. of a paraffin could be converted into a mixture of fatty acids, aldehydes, alcohols, etc. Schreyer and Kragen, on the other hand, obtained small yields of naphthenic and polynaphthenic acids by passing a slow stream of dry air through petroleum distillates (specific gravity ·7844 to ·8200) at the boiling point, the catalyst being 5% of finely powdered caustic soda and a few drops of mercury. In a French patent, Wohl described the catalytic effects of platinum and palladium. Schulz, working without a catalyst, produced aldehydes, asphalt and petroleum acids from petroleum hydrocarbons heated to 300°C. By blowing air through Galician paraffin (M.P. 52°C.) at 130-135°C. for 15 to 17 days, Beryman obtained a brown reaction product (B.P. 132°C.) from which he isolated formic, acetic, normal butyric and lignoceric acids together with two solid acids of formulæ $C_{11}H_{22}O_2$ and $C_{16}H_{32}O_4$. A German textile firm found in 1918 that the oxidation of paraffin with air, but without a catalyst, at 130°C., gave after 72 hours a darkish, malodorous product with a saponification number of 53. With the addition, however, of finely divided pyrolisite or manganese stearate, much better results accrued. Thus, with only 1% of manganese stearate, 83% of the reaction product was almost white and possessed a saponification number of 198. The product resembled coco-nut oil in smell and appearance and consisted of volatile fatty acids.

Frank found that fatty acids were formed during cracking in presence of nascent oxygen and catalysts. Yields of 20 to 33% of fatty acids were produced from paraffin after several hours' treatment at 153°C. For complete oxidation, 10 to 30 hours' treatment was essential, whereas with lead as a catalyst, the time could be reduced to two to four hours. The product had a saponification number of 250-353 and an acid number of 220-230. Fractional distillation of the ethyl ester gave 18·75% volatile to 253°C. and 78·1% of higher boiling constituents. The former fraction

contained acetic, propionic, butyric, valeric and caproic acids, and the latter solid fraction contained myristic, palmitic, stearic and arachidic acids. Frank maintains that catalysts have an effect on the chemical composition of the product. W. S. C.

391. Fuel Technology in France. D. Brownlie. *Engineer*, July 6, 1928, 18-19.

Strenuous efforts are being made to render France independent of foreign sources of fuel oils and coal by developing water power, encouraging the use of materials other than petrol (*e.g.*, producer gas and alcohol) for driving motor vehicles, producing synthetic liquid fuels and adopting improved methods for the combustion of solid fuels. With regard to synthetic fuels, the trend is not so much in the direction of hydrogenation as in the catalytic reduction of carbon monoxide. Low-temperature carbonisation is in a disappointing state, although two large Salerni retorts, each having 17 troughs, are now in successful operation in the Sarre. Other processes, but about which little is known, are the Bonnevie, Dupuy, Guardabassi-Gouillard, Migeon and Pieters, most of which relate to the treatment of lignite or non-coking coals. The French shale-oil industry continues, especially at Autun, with a total consumption of about 200 to 250 thousand tons of shale per annum. H. G. S.

392. Distillation Test and Vapour Pressure. G. G. Oberfell, R. C. Aldur and L. H. Fitch. *Oil and Gas J.*, May 31, 1928, 148.

It has been proposed to use the I.B.P. in the A.S.T.M. distillation test as an indication of the vapour pressure of gasolines. In the present work, actual determinations of vapour pressures were made by the Busth-Prather method, and these were compared, firstly with I.B.P.'s, with no definite success, and then with the temperatures of the 5% distillation point in the A.S.T.M. method. Due to the high losses, however, it was found that the distillation curve had to be exterpolated to give corrections for the initial loss.

Tables are given from the data obtained, by the use of which it is claimed fair approximation to the actual vapour pressure may be obtained from the A.S.T.M. distillation. A. R. S.

393. Fundamental Principles in the Design of Gas Burning Equipment. N. T. Branche. *Chem. and Met. Eng.*, 1928, xxxv, 357.

This paper describes the means by which the area of burner nozzles and the type of inspirator necessary for burning air-gas mixture may be determined.

A graph is given showing the relation between the gas discharge per square inch of nozzle area and the pressure on the gas mixture, for three gases, blue water gas (310 B.Th.U.), natural gas (967 B.Th.U.) and an artificial gas (530 B.Th.U.).

The lower limit to which a burner may be turned down depends on the type of gas employed owing to the danger of firing back if the mixture pressure at the nozzle falls too low. The upper limit is determined by the pressure which it is practicable to apply to the mixture. The lower limits of pressure are given as ·5 inch water for a gas rich in hydrogen, and ·2 inch for natural gas.

The devices described for the purpose of keeping a correct proportion of gas to air depend on the entrainment of one fluid by the other in a Venturi meter. The gas may be fed to the meter under pressure and made to entrain the air; or the reverse procedure may be adopted, in which case the gas must be brought to atmospheric pressure by means of a governor.

Types of equipment used in various commercial set-ups are described.

P. D.

394. Improvements in or Relating to the Refining of Liquid Hydrocarbons. Silica Gel Corpn. E.P. 292,231, 1928.

Unrefined oils may be freed from sulphur and gum forming compounds by treating in air percolator systems at temperatures from 250°—300°F. with silica gel or other porous substance of porosity such that it is capable of absorbing at least 21 % of its weight of water at 30°C. when it is in contact with water vapour at a partial pressure of 22 m.m.

The gel used may be unimpregnated, or impregnated with some metallic oxide capable of easily combining with sulphur, the maximum amount of oxide used being 1% by weight. Sulphuric acid of 66° Be. strength may be used in addition, either to the oil before percolation or else in impregnation on the gel. The use of sulphuric acid causes precipitation of the gum-forming constituents, the amount of acid used being 0·1 to 0·5 per cent. When the acid is added to the oil before impregnation, the total amount may be added to the first 20% of oil, the gum deposited on the gel by this addition being capable of promoting the formation of gums from the remaining 80% of oil.

A. R. S.

395. Improvements in or Relating to Temperature Controlling Devices for Furnace Chambers and Other Apparatus. N. H. Freeman. E.P. 282,864, 1927.

This device consists of a valve placed in the fuel supply line which is controlled by means of a solenoid. The solenoid is energised by means of an electrical device dependent on the temperature (such as described in Pat. Spec. No. 174,024).

The valve consists of a pair of ported sleeves open at their lower ends, the inner sleeve sliding on a vertical axis.

An adjustable stop is provided for limiting the degree of movement of the valve.

P. D.

396. Improvements in Colorimeters. C. Zeiss. E.P. 280,552, 1928.

This colorimeter is designed for the quantitative determination of colouring matter in a solution. The two sides of the colorimeter are the same in construction and are traversed by pencils of light obtained from the same source. Its use depends on the measurable variation of the depth of liquid through which the light is allowed to pass. Each side of the system is made up of two cylindrical containers with glass bottoms, through which the light is allowed to pass, and a colourless glass rod, all mounted concentrically on a vertical axis. The diameters are such that the glass rod, which is mounted at the top, slips into the central container, while the latter slips into the lower container. The rod and lower container are in fixed positions, while the central container is capable of vertical movement, the extent of movement being recorded on a scale. When this container is moved into the highest possible position the liquid in it is totally displaced by the glass rod and the beam of light passes through the total length of the liquid column in the lower container. When the central container is lowered to the fullest possible extent, the liquid is completely displaced from the lower container and the light passes the total length of liquid column in the central container. Intermediate positions make it possible to mix the colours due to the two containers in all proportions.

By choosing the correct solutions for filling the four containers, it is possible to obtain a true colour match and to estimate the quantity of colouring matter in a solution from comparison with the pure solvent and a solution of colouring matter of known concentration.

P. D.

397. Effect of Alkali-Metal Compounds on Combustion. C. A. Thomas and C. A. Hockwalt. *Ind. Eng. Chem.*, 1928, **20**, 575.

Dilute aqueous solutions of alkali metal salts have been found to be effective in combating oil and wood fires. With the exception of rubidium, the efficiency of the metal for the purpose increases with rise of atomic weight, while the influence of any particular radical is the same for all alkali metals. The presence of oxygen in the compound used increases its efficiency. For example, potassium chlorate is two-fifths more effective than the chloride, while the perchlorate is five times more efficient than the chloride. The strengths of solutions required to show positive effects in extinguishing oil fires are as follows, the figures denoting normality. Lithium acetate 5·0; sodium dichromate 2·0; chloride 4·7; acetate 1·0; potassium dichromate 0·2; chloride 1·0; perchlorate 0·2; acetate 0·7. A. R. S.

398. Improvements in Flow Regulators. C. H. Smoot. E.P. 271,814, 1928.

The improvement consists of a device for controlling the rate of flow of two or more fluids which are required to be mixed in a definite proportion throughout the range of flow. The device consists of a chamber to which air under pressure is delivered from a supply pipe. The pressure in the chamber is controlled by means of a relief-valve which may be made to operate by a mechanism influenced by some external source upon which the rate of flow of the fluids will have some effect (*e.g.*, in the case of oil and air being supplied to a burner, the valve may be made to operate by means of the steam pressure in the boiler). The air leaves the chamber by means of conduits, one for each liquid under control. The admission of air to and from the conduits is controlled by valves, and by suitable setting, the pressure in the conduit may be maintained at any fixed proportion of that in the chamber, and will follow this proportion as the pressure in the chamber varies.

The pressure from the conduit acts on a diaphragm connected with the control valve in the flow line of the fluid. The movement of the valve is also influenced by a second diaphragm actuated by pressure directly due to the fluid flowing in the pipe (*i.e.*, pressure connections taken from either side of an orifice in the line). Thus the efforts of the chamber to open the valves are counter-balanced by forces proportional to the square of the fluid velocity.

Safety valves are fitted to the air pressure chamber for dealing with abnormal conditions arising in the flow lines. P. D.

399. Improvements in or Relating to Thermocouples for Pyrometers. Electroflo Meters Co., Ltd. E.P. 288,105, 1928.

An enclosed head is provided for the thermocouples in such a way that the couples and leads are fully protected from the atmosphere. The head contains two terminals to which the couple and heads are connected. For the purpose of making this connection, and for adjustment after the couple and lead conduits have been fixed in position, a plate in the head may be removed. This plate is held in by screws and is rendered gas-tight by means of a washer.

The couple sheath and the tubular metal conduit for the leads screw into the head. P. D.

400. Improved Mixture for Use in the Construction of Cold Asphalt Road Pavements. A. C. Brown. E.P. 290,725. Feb. 17, 1927.

In the manufacture of road material a liquefier, to which a small amount of high penetration bitumen has been added, is used. Bitumens of varying penetrations, mixed to give a penetration of 65 to 80, may be added to the aggregate. The bitumen may be heated before being placed on the aggregate.

In the example quoted, the road surface is laid in two coats. For the base coat the broken stone is heated to over 212° F. to dry it and then cooled to 100° F. It is then sprinkled with 0.3 per cent. by weight of kerosine containing 1 in 400 parts by weight of 200 penetration bitumen. The kerosine has the following specification:—Sp. gr. 0.810; flash-point, 110° to 115° F.; I.B.P., 150° C.; F.B.P., not over 300° C. When all the stones are coated, 5 per cent. by weight of approximately 75 penetration bitumen is added at 250° F. The whole is then sprinkled with 0.4 per cent. of finely-ground hydrated lime followed by 2.75 per cent. of stone dust.

The top coat is mixed in a similar way, but with the following proportions:—Bitumen, 6.5 per cent.; kerosine-bitumen solution, 0.6 per cent.; lime, 0.6 per cent.; stone dust, 3.3 per cent. G. S.

401. Composition for Use in Substitution for Asphalt and Bituminous Materials and for Electrical Insulation. G. T. Fuery (from Australian Bitumenous Compounds, Ltd., Melbourne, Victoria). E.P. 290,886. Sept. 20, 1927.

A substitute for asphalt or bitumen is claimed which consists of a mixture of oil-shale and tar, with which may be incorporated a suitable filling material. An excellent road material consists of 80 per cent. unburnt ground limestone and 20 per cent. of the basic material, while a mixture containing 80 per cent. shredded waste rubber forms a resilient paving composition. For insulation purposes the basic material may be used alone or incorporated with a suitable dielectric substance. G. S.

402. Treating Emulsions. C. F. Pester, Assr. to Standard Oil Development Co. U.S.P. 1,669,379. 1928. Appl. 20–3–1923.

A method is here described for breaking emulsions of hydrocarbon oils and water. Unneutralised sludge obtained by refining hydrocarbon oils with concentrated or fuming sulphuric acid, is added to the emulsion and the mixture heated to 100°–212° F. or not as desired. The proportion of sludge used varies in general from ¼–5 per cent. according to the emulsion concerned, more resistant emulsions requiring more of the sludge. The mixture on standing separates into an upper oil layer and a lower layer of water and dirt, which are then separated. H. E. T.

403. Treating Emulsions. L. Burgess, Assr. to Standard Oil Development Co. U.S.P. 1,668,941. 1928. Appl. 20–3–1923.

A method is described for treating emulsions of hydrocarbon oils and water. Sulphuric acids derived from the sludge of hydrocarbon oils accomplish an improved result in breaking emulsions. Sludge separated from an oil, which has been treated with strong sulphuric acid, is purified by repeated washing with dilute hydrochloric acid to obtain the sulphuric acids. From ¼–5 per cent. by volume of the sulphuric acids is then added to the emulsion and the mixture heated to 100°–212° F. to accelerate separation. On standing, the mass separates into two layers, the upper one being oil and the lower dirt and water, which can be easily separated. H. E. T.

404. Demulsifying Hydrocarbons. G. Egloff. U.S.P. 1,670,107, 1928.

This is an improved process for dehydrating and cracking emulsified oil under heat and pressure or vacuum in the presence of an electrolyte such as caustic soda. A suitable apparatus consists of a still composed of a series of vertical pipes expanded into upper and lower drums and set in the Dutch oven part of a furnace. A conduit from the upper drum leads to a condenser

and receiver. Interposed between the drums is a circulating pump and discharge control valve. Valves regulate the entry of raw oil and electrolyte, and a spray device is provided for distributing them. In one method of operation, the still is charged by means of the pump and pressure is maintained through the entire system whilst a given quantity of caustic soda is simultaneously admitted. While vapours pass through the conduit, the circulating pump is in action, demulsification and cracking taking place simultaneously. Instead of pressure a vacuum may be used in which case cracking is substantially avoided. The use of an electrolyte obviates the tendency to foaming. H. S. G.

Chemistry of Petroleum.

405. THE OXIDATION OF n-HEXANE. M. Brunner and E. K. Rideal. *J. C. S.*, 1928, 1162—1170.

In order to study the process of oxidation of n-hexane, known mixtures of hexane and oxygen were sealed in a glass tube connected to a mercury manometer. The bulb was heated during some 90 minutes to an observed temperature (210° to 240° C.). In the first series of experiments, mixtures of varying composition were heated until the sudden rise in pressure indicated that initial combustion was taking place. With all hexane mixtures used, the temperature of initial combustion (T.I.C.) was noted at 232° C.: the following observations were also made.

Hexane % by volume.	% *increase in* press. at T.I.C. = b.	% *Oxygen* b	Gas Analyses.			
			CO_2 .. Unsats...	CO ..	O_2
77·9 ..	3·39 ..	6·5 ..	– .. – ..	– ..	–
63·3 ..	5·53 ..	6·6 ..	– .. – ..	– ..	–
50·8 ..	7·28 ..	6·7 ..	28·04 .. ·23 ..	27·73 ..	–
35·7 ..	12·0 ..	5·3 ..	– .. – ..	– ..	–
33·7 ..	11·7 ..	5·7 ..	38·04 .. 1·13 ..	29·76 ..	0·36
31·7 ..	Explosion ..	— ..	7·01 .. 3·61 ..	26·65 ..	2·5

In the second series of experiments, various hexane mixtures were heated to a steady temperature and the time of induction of the reaction was noted. The following table indicates the results.

Hexane % by Vol.	Final Temp. of Oil BBth.	Apparent period of Induction (mins).
50·8 ..	232° C. ..	nil
45·7 ..	220° C. ..	7
45·2 ..	215° C. ..	17
46·1 ..	210° C. ..	37
43·8 ..	210° C. ..	30 (*a*)
46·0 ..	210° C. ..	232 (*b*)
44·0 ..	210° C. ..	76 (*c*)

(*a*) Final pressure increase = 86 m.m. shewing the influence of pressure on shortening the indication period.

(*b*) 2·0 m.g. of aniline present, final pressure increase only 31 m.m.

(*c*) 0·25 m.g. of aniline present, final increase = 73 m.m.

The existence of the period of induction is very well marked. When the mixture is passing through the period of induction, a slight decrease in pressure,

some 15 m.m., is observed. This may be due to the formation of a moloxide and of the peroxide and also of polymerised products of unsaturated bodies. In a further experiment, the mixture was suddenly chilled and examined after it had almost passed through the period of induction. Of the original hexane, 3·2 per cent. was found to be present as the peroxide. Another experiment showed that after a 35 minutes induction period, at 210° C., 30 per cent. of the original oxygen had been consumed.

When powdered pumice was present, combustion took place at a slow rate and no sudden rise in pressure, even after 325 minutes, was observed. During this slow combustion, hexane peroxides decompose to aldehyde and water, followed by further partial oxidation and condensation of the aldehyde formed: the peroxide also reacts with hexane to produce unsaturated hydrocarbons and water.

The authors suggest that the reactions involve the formation at catalytic surfaces of an active mo/oxide which is subsequently converted to a more stable peroxide. The peroxide can react either in the gas phase or at the surface, but when the peroxide concentration reaches a critical value, chain propagation ensues causing excitation and combustion of molecules coming in contact with the reaction centres of the chain. W. N. H.

406. CATALYSTS FOR THE FORMATION OF ALCOHOLS FROM CARBON MONOXIDE AND HYDROGEN. K. Frolich, M. R. Fenske, and D. Quiggle. *Ind. Eng. Chem.*, 1928, **20**, 694—697.

This paper is the first of a series dealing with studies on the fundamental nature of catalysts employed in the synthesis of methanol from carbon monoxide and hydrogen. Considerable information has already been obtained concerning the synthesis of methanol using high pressure and different catalysts (*Ind. Eng. Chem.*, 20, 285). It was deemed advisable to investigate the reverse reaction—*i.e.*, the decomposition of the alcohol—in the presence of the same type of contact substances.

A study of the decomposition permits operation at atmospheric pressure, it being justifiable to suppose that the catalyst giving the maximum decomposition into carbon monoxide and hydrogen will be particularly suitable for the synthesis of methanol under pressure. This paper is limited to a discussion of catalysts composed of copper and zinc.

The catalysts were prepared by adding ammonia to a mixture of zinc and copper nitrates dissolved in water. The method of precipitation—zinc and copper hydroxides together—was chosen because other methods showed that the resulting mixtures were inferior in activity.

The apparatus used for the experimental work is described. Roughly, it consists of a vapouriser, an electrically heated reaction tube, condensers and gasometer.

The constancy of the reaction rate over the whole period of individual runs is shown. In only a few cases is there a slight deviation from the straight-line course.

The free energy change for the decomposition of methanol into carbon monoxide and hydrogen is

$$\underset{(1\text{ atm})}{CH_3OH(g)} \rightarrow \underset{(1\text{ atm})}{CO(g)} + \underset{(1\text{ atm})}{2H_2(g)}$$

$$\Delta F = 20{,}480 - 43{\cdot}1T \log T + 0{\cdot}01545T^2 + 69{\cdot}8T$$

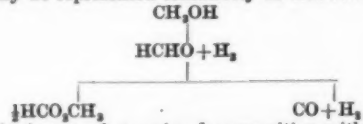
From which ΔF at 0°C. is found to be 12,070 calories and ΔF at 363°C —5,530 calories. A calculation of the equilibrium shows that at 360°C. and a total pressure of 1 atmosphere, the methanol is about 99 per cent. decomposed.

The rate of flow of methanol vapour over the catalyst was chosen to give a maximum decomposition of about 50 per cent. in order to investigate the activity of catalysts at conditions sufficiently remote from equilibrium. A small amount of ZnO—*e.g.*, 3 mol per cent.—will increase decomposition three-fold. When a small amount of copper oxide is added to zinc oxide, action is also apparent; —3 mol per cent. increases decomposition only 30 per cent. Over the range 15 to 45 mol per cent. zinc oxide, the decomposition remains practically constant. At the point where zinc oxide becomes present in excess, however, there is a small, but noticeable, increase in decomposition.

It must be remembered that the decomposition is a true measure of the catalyst only when the time of contact and concentrations are the same in each case. The products of reaction are constantly diluting the reaction constituents, therefore the velocity is continuously increasing over the length of the catalyst, *i.e.*, the time of contact is decreasing. The result is a flattening of the peak of the decomposition curve, making the most active catalyst stand out less markedly than it would have done had the time of contact been constant.

Examination of the products of the reaction shows that the decomposition is not a simple reaction. Formaldehyde, methyl formate, carbon dioxide, and methane are formed in addition to carbon monoxide and hydrogen.

The information obtained regarding the mechanism of the decomposition will be the subject of the second paper in the series. For the present the decomposition may be represented tentatively as follows:—



The evidence is in favour of stepwise decomposition with formaldehyde as an intermediate product.

The extent to which decomposition takes place depends greatly on the zinc-copper ratio of the catalyst. Carbon monoxide and methyl formate together with an equivalent amount of hydrogen are the principal products, the addition of a small amount of copper to zinc oxide favouring the formation of carbon monoxide and of a small amount of zinc oxide to copper, the formation of methyl formate. The carbon-monoxide curve is in accordance with statements made relating to the synthesis of methanol, that the more basic oxide should be in preponderating quantities. The carbon-monoxide curve takes an enormous rise between 40-50 mol per cent. zinc oxide, the maximum occurring about 70 per cent. A catalyst which carries decomposition down to carbon monoxide is also a good catalyst for the synthesis of methanol by high-pressure reduction of carbon monoxide. Further evidence will be presented in subsequent papers. Further work under way consists of determining the chemical composition of the spent catalysts supplimented by an X-ray investigation of their physical structure. Similar studies are being made of the binary system zinc-chromium, and finally the data obtained on decomposition will be correlated with activity determinations on the synthesis of methanol. R. P.

407. The Rôle of Catalysts in High-Pressure Synthesis from Water-Gas. P. K. Frolich. *J.S.C.I.*, 1928, **47**, 173-179T.

The author reviews the known reactions of water-gas and bases the problems of high-pressure synthesis upon the production of ammonia from its

elements. The possibility of building up practically any aliphatic organic compound from carbon monoxide and hydrogen was first foreshadowed in German Patent 293,787 of 1913, and was followed ten years later by Fischer's synthesis of "synthol." As in the case of ammonia, catalysts are required, but the endless number of possible reactions calls for catalysts of an unusually selective nature. The reactions are also extremely sensitive to heat, requiring rigid temperature control, whilst a change in pressure may lead to a profound alteration in the mechanism of interaction. The methyl-alcohol process was the first tangible result of the efforts to reduce the complexity of the products and was made possible by the development of the proper catalyst, *e.g.*, two or more metallic oxides, the number of possible combinations being almost unlimited. It is calculated that the heat evolved at optimum conversion is approximately eight times that liberated for the same volume of gas entering a Haber ammonia unit, necessitating either the dissipation of relatively large quantities of heat or a reduction in the conversion per passage. The linking of two or more carbon atoms is a complicated process. The iron-alkali catalyst successfully brings about this reaction but its action is too violent and better results are attained by the addition of an oxide of the alkali or alkaline-earth metals. Whilst temperature, pressure and rate of flow may be of importance in the synthesis of alcohols higher than methyl, the character of the catalyst is all-important, no satisfactory explanation of the reactions has yet been given. It is known that zinc oxide, when mixed with copper oxide, in the preparation of an alcohol catalyst, is reduced to a lower state of oxidation, or more probably to metallic zinc, this change being traceable by analysis of the catalyst or by the carbon dioxide content of the reducing gas. Conversely, metallic oxides which are known to be easily reducible do not suffer complete reduction when present in such mixtures as are used in the synthesis of alcohols. The necessity of having the more basic oxide in excess is repeatedly emphasised in the patent literature.

The paper concludes with data relating to the decomposition of methyl alcohol and the effect of variations in the composition of a zinc oxide—copper oxide catalyst, the nature of the products being shown in graphs.

H. G. S.

408. Improvements in and Relating to the Conversion of Saturated Hydrocarbons, in Gas or Vapour Form, especially Methane, into Unsaturated Hydrocarbons. J. G. Farbenind, A.G. E.P., 264,845, 1928.

Gaseous hydrocarbons, especially methane, are diluted with nitrogen or other inert gas and passed through a flame, preferably a disc-like flame of high temperature more or less completely filling the cross sectional area of the tube. The products contain acetylene, which is separated by suitable means or is transformed into such compounds as acetaldehyde, benzene and the like. A portion of the original gas may be employed to feed the flame and the apparatus is designed so that the mixed gas may be preheated by the hot gaseous products.

H. G. S.

409. Improvements in the Manufacture and Production of Liquid Hydrocarbons of Low Boiling Point From Olefines. I. G. Farbenind, A.G. E.P. 291,137, 1928.

Liquid hydrocarbons of low boiling point are produced from olefines or gases containing olefines by heat treatment under any suitable pressure in presence of catalysts. The latter may be gold, silver or copper in such form as to exhibit coherent surfaces, compounds, preferably oxygen-containing compounds of the alkali metals, salts of the oxy-acids of phosphorus,

boron or antimony with alkali or other metals as the basic constituent, or the free oxy-acids, when solid, of phosphorus, boron or antimony. When working under pressure with a gas containing both hydrogen and carbon monoxide, catalysts capable of forming methanol must be excluded. The most suitable temperatures generally range between 200° and 700° C. At low temperatures, *e.g.*, 300° to 400°C. the products usually contain large amounts of liquid olefines, but at about 550°C. the bulk of the products consists of benzene hydrocarbons and naphthenes in varying proportions.

H. G. S.

410. Production of Olefines. A. W. Nash, A. R. Bowen and O. C. Elvins. E.P. 291,867, 1928.

The production of olefines is effected by passing a mixture of carbon monoxide and hydrogen, the former being in excess, over a catalyst consisting of a reduced mixture of the oxides of cobalt, copper and manganese, preferably in the granular state. The catalyst is maintained at from 280° to 300°C., and the pressure at atmospheric. In the first of two examples given, a yield of 3·6% of olefines and 6·6% of saturated paraffins was obtained by passing a mixture of carbon monoxide (55·7%) and hydrogen (43·4%) over a cobalt oxide—copper oxide—manganese oxide catalyst maintained at 286°C. the catalyst being enclosed in a tube of approximately 1·5 sq. cm. through which the gas was passed at a speed of 4·2 litres per hour and at atmospheric pressure.

H. G. S.

411. Purification of Aromatic Hydrocarbons. The Seldon Co., Pittsburg, Pa. E.P. 290,840. 1927.

The object of the invention is to replace the wasteful treatment of crude benzoles with sulphuric acid and alkali washings, and to remove in one step not only basic and unsaturated impurities, but also organic and inorganic sulphur compounds, phenols and aliphatic hydrocarbons in a form in which they can be utilised to produce resins, solvents and other bodies. The process consists in subjecting the crude aromatic hydrocarbons, with or without limited sulphuric acid treatment, to selective halogenation with chlorine or bromine or suitable bodies that yield them, with or without halogen carriers, under suitable conditions. The aliphatic compounds, saturated and unsaturated, thiophene and other sulphur compounds, and phenols and basic compounds are preferentially attacked, and form for the most part high boiling compounds from which the aromatic hydrocarbons can be distilled off in a highly purified state. Elemental chlorine, gaseous or liquid, may be used alone or in solution, or bodies yielding chlorine such as aqua regia, phosgene, sulphuryl chloride, nitrosyl chloride, other relatively unstable chlorides, hypochlorites, etc., or hydrochloric acid in the presence of an oxidizer. Similarly bromine or brominating agents, or mixtures of chlorinating or brominating agents may be used. Iodine, however, does not act satisfactorily as a halogenating agent, although it is an effective halogen carrier and may be used as such. Any of the known halogen carriers may be used singly or together to facilitate the reaction. The reaction may advantageously be carried out in the presence of light, heat, pressure, silent electric discharge or catalysts which facilitate the desired reactions. The purification may take place in the vapour phase, and the reaction products fractionally condensed. Alkaline compounds may be added in small quantities to neutralize any acid given off. In some cases it may be advantageous to combine a modified acid wash with selective halogenation in order to remove undesired impurities in precisely the form most suitable for subsequent use. Numerous specific examples are given of the general process described. H. S. G.

Crude Oil.

412. Process for Distilling Mineral Oil. A. E. Pew and H. Thomas. U.S.P. 1,668,602, 1928. Appl. 23-2-1924.

A process is described for the fractional distillation of crude oil into gasoline fractions of various grades, turpentine substitutes or kerosine, or into fractions of progressively increasing boiling points. The apparatus consists of a number of stills and towers arranged in series, the number depending upon the number of desired cuts. H. E. T.

413. Distillation of Liquid Mixtures. J. P. Fisher. U.S.P. 1,672,978, 1928.

This invention relates to the distillation of liquid mixtures and in particular to distilling a petroleum oil, and is an improvement on the process and apparatus described by Henry L. Doherty in Patent No. 1,472,116, 1923.

The Doherty process consisted of passing oil through a series of zones of progressively increasing temperature, the temperatures being maintained by a contrary stream of initially hot gas. The oil vapours from each zone are condensed and removed after passing through a condensate zone intermediate between the oil zone from which the vapour was obtained and the next oil zone at a higher temperature.

The present invention provides many improvements over the Doherty process although following the same general principles. The charging stock is introduced at the top of a column still and flows round a coil in the top thereof before passing to another coil below the top baffle. In this latter coil, the oil is exposed to vapours and counter-current gas rising from the compartment below at a somewhat higher temperature. Some vapours condense on the coil and drip into a collecting basin, having a vapour trap and discharge pipe to the outside of the column. The oil passes from this condensing coil into the space between two shallow conical plates, the lower surface lying just above the coil and acting to supply more condensate to the collecting basin. The gas and uncondensed vapours pass upwards through a central short pipe through the two conical plates and then again upwards through a perforated bubble plate. Oil from between the cones is caused to flow up another pipe to near the centre of the bubble plate, whence it flows around a spiral baffle until it reaches the edge where an overflow pipe is situated. The overflow pipe passes downwards to a point below the second baffle where another condensing coil and compartment generally similar to the first is located. This cycle of operations is repeated down the column until the bottom is reached where the overflow pipe discharges into a shell still fixed to the bottom of the column and heated by a furnace. In the apparatus described eleven bubble plate and condensing compartments are used.

It will be understood that the lighter fractions present in the oil are vapourised at any bubble plate and the vapours pass up to the cooler zone where they first meet the limited area of a condensate collecting basin. A small amount of vapour is condensed here and runs back as reflux, while the main portion of vapours are condensed on the condensing coil a little higher up, any excessively light vapour passing to the compartment yet higher up.

From the top of the column the vapours and circulated gas pass to a condenser where all vapour except that necessary to saturate the gas is removed. The gas passes to a blower and is pumped back to a perforated pipe disposed in the bottom of the shell still below the column. The gas is heated by bubbling through the hot residue oil and again passes to the column. Residue

oil is continuously run off from the bottom of the shell still while charging stock is also supplied continuously to the top of the column.

To supply heat to balance that lost through drawing off hot condensate from the column the whole column is enclosed in a jacket and the flue gases from the furnace of the shell still are constrained to pass upwards through the annular space thus formed. E. R. B. S.

414. Apparatus for Distillation. H. M. Godsey. U.S.P. 1,673,238, 1928.

This invention relates to the distillation of liquids which will not appreciably absorb the products of combustion from a furnace and are not appreciably damaged or contaminated by contact with the same. It applies more particularly to the distillation of petroleum oils.

The apparatus described admits of heating oil in a still by direct contact with the furnace gases and thus avoids many disadvantages and dangers due to the usual method of heating indirectly through the metal wall of a container.

Combustion of oil or gas fuel takes place in a well-built furnace so arranged that only sufficient air for complete combustion may be admitted. The furnace gases pass through a stack in which is a pipe for the admission of steam or inert gas, if desired, for diluting or reducing the temperature. The hot gases pass thence into suitable spraying device in the bottom of a still where they are allowed to bubble up through the oil contained therein and heat it by direct contact. The vapours and gases then pass through a fractionating column having an oil supply pipe at the top, and on to a condenser where the vapours are liquified. The liquid is removed through a trap or barometric column while the gases pass on to a gas exhausting apparatus and are discharged.

The plant is intended to work under reduced pressure and by the addition of a residue run off from the still, may be made to work continuously. E. R. B. S.

415. Process for Distilling Emulsified Oils. G. Egloff and H. P. Benner. U.S.P. 1,674,819, 1928. Appl. 24-4-1928.

By means of a system of steam coils the emulsified oil is subjected to a "surface distillation" at atmospheric pressure. The steam coils are arranged in layers which are independently operated and controlled; in this way the heat necessary to distil off the water from the oil can be applied. With this system of steam coils, the point of application of the heat in the still can be progressively lowered, as the oil level falls during the progress of the distillation.

When the percentage of water remaining in the oil reaches a desirable low value, the distillation may be completed by the application of external heat to the still. H. E. T.

See also Abstract No. 429.

Motor Spirit.

416. Essential Treating Details for the Hypochlorite Method. W. R. Hounsell. *The Refiner*. 1928, 4, 63.

The following treatments are used on benzine from high sulphur crudes and on intermittent treating. "Recovered Soda" refers to caustic soda recovered from previous treatments in small stills. The benzine in the

agitator is given a 3 per cent. treatment with 10° Bé recovered soda agitated by circulation through a centrifugal pump for 30 minutes, allowed to settle and the spent soda drawn off. The oil is then given a 20 per cent. wash with water, settled, and the water drawn off. Then comes the 16 per cent. hypo treatment, agitation for 2½ hours, the settling and drawing off. Finally the batch is given a 3 per cent. treatment with 10° Bé fresh caustic soda and settled. This soda is not washed out with water, but the oil goes to the continuous benzine stills while still holding some of the solution in suspension. The final permanganate test after treating should be about 30 seconds. After this treatment, the residual normality of the spent hypo should be zero, and if much sulphur is present in the untreated oil, it usually requires two such treatments with hypo in succession, before the final soda wash. In this case the normality of the first waste hypo should be zero, and that from the second treatment about 0·09N. This solution from the second wash can then be made up to strength by the addition of strong hypo from storage and used again for the first treatment.

Gasoline usually receives only one agitation with hypo provided the benzine has been previously hypo treated, but if the benzine has received only a caustic soda treatment and a water wash, then two hypo treatments are given to the gasoline. Gasoline for one agitation requires 13½ per cent. of 0·34N. hypo. Gasoline for two agitations requires 16 per cent. and 9 per cent. respectively of 0·34N. hypo. The gasoline first receives the hypo treatment, this being continued until the normality of the hypo is about 0·05; at this stage the gasoline must pass a 5-minute permanganate test. Next comes a 3 per cent. wash with 13° Bé caustic soda for 30 minutes, and finally two 20 per cent. water washes lasting 10 minutes each.

Where calcium hypochlorite is the reagent used, it occasionally happens that a little "medicine" is required to bring the action to completion. Suppose, for example, after considerable agitation the normality of the hypo refuses to go below 0·2. This condition can be remedied by adding 14–25 lb. of sodium bicarbonate, which liberates the nascent oxygen and combines with the chlorine. Ferrous sulphate dissolved in hot water has the same apparent effect.

A continuous treating plant operates as follows. The untreated benzine enters the suction of a centrifugal pump on agitator No. 1, mixes with the soda already in the cone of the agitator by the impellers of the pump, is pumped over the top of the agitator, down through a pipe 8 ft. 6 in. long, and strikes the baffle plate attached to the end of the pipe, where further mixing takes place. The overflow of benzine from agitator No. 1 enters the suction of agitator No. 2 pump, mixes with water from the cone of the agitator, and is thrown over the top into agitator No. 2. In agitator No. 3 the washing is a repetition of that in No. 2. The hypo enters the benzine in agitator No. 5, from a measuring box containing a "V" notch device, and gravitates to the cone. It then mixes with the overflow from agitator No. 4 in pump No. 5, and is taken back over the top of the agitator No. 5. A little of this mixture is allowed to flow into the top of agitator No. 4, to keep it supplied with a weak solution of hypo, and this solution which flows back is reinforced if necessary with strong hypo from storage. Pump No. 4 delivers a mixture of weak hypo and benzine over the top of agitator No. 4, and enough of this is allowed to flow into a small separator from which the hypo runs down to a waste box. The overflow from agitator No. 5 runs to pump No. 6 and receives a final soda wash, is pumped back over the top of agitator No. 6, and then overflows to the finished benzine tanks. These operations are run in counter-current treatment, the oil running through the system in the opposite direction from the flow of chemicals. Thus the raw benzine meets the weak solutions and leaves the system under the influence of fresh solutions. S. W. C.

417. Treat Cracked Distillate with Acid in Continuous Operation, W. R. Hounsell. *Oil and Gas J.*, 1928, **27** (3), 201, 205.

The object of the treatment of cracked distillates is to remove colour, odour, gum-forming and corrosive compounds, and to stabilise against reversion by storage or sunlight. As far as is known, these objectionable properties are due to hydrogen sulphide, organic-sulphur compounds, alkyl-sulphides, mercaptans, diolefines, acetylenes and nitrogen compounds.

While straight-run distillates consist chiefly of paraffins, cracked distillates are largely composed of aromatics, naphthenes and olefines. The aromatics and olefines may be sulphonated, oxidized or polymerized by the acid, but must be preserved, as they are responsible for the superior power and anti-knock qualities of cracked motor fuels. They also have higher carbon-atom concentration than the paraffins, and therefore higher B.Th.U. value.

The continuous system of treatment is best suited to cracked distillates, but the biggest problem is the design of equipment. The use of 66° Bé gives high losses, and experience indicates that 62° to 64° Bé acid, or about 84 per cent. H_2SO_4 is the best strength. This strength re-acts with di-olefines and sulphur compounds fairly rapidly, does not react noticeably with aromatics, and reduces reaction with the olefines to a minimum. It does not attack iron or steel, and is not so difficult to reclaim from acid-sludge. The mixing of acid and distillate should be done by mechanical means. The time required to secure the necessary contact is the only time factor.

In one installation the acid used was 62° Bé, the distillate from the cracking system being 49° Bé, containing 50 per cent. aromatics and olefines. After two washes with equal quantities of water, the distillate was allowed to come to equilibrium in storage. It was then passed to the treaters with the requisite amount of acid, in this case 5 lb. per barrel. From the treater the oil and acid then passed to the first settling shell, the overflow from this passing to a second settling shell. After this, the oil was given two water washes and delivered to the alkaline treatment, which may be the usual doctor or a more active solution. G. S.

418. Liquid Fuel for Use in Internal-Combustion Engines and Process for the Production thereof. P. T. Somerville and W. H. Hoffert. E.P. 289,347, 1928.

The process is designed to prevent resinification of a proportion or all of the unsaturated bodies which tend to form either by oxidation or polymerization in cracked spirits and benzoles.

Attempts to remove the less stable unsaturated hydrocarbons by selective means having proved unsuccessful, the authors suppose that stability of some cracked spirits is due to the presence of inhibitors and to the absence of gum-forming accelerators. They have shown, by gum tests before and after exposure to ultra-violet radiation in glass vessels, that the gumming is substantially reduced by the addition of definite small amounts of aromatic phenolic, nitro and amino compounds. There seems to be a maximum amount of compound which may not be exceeded. In the examples given tricresol is suggested as satisfactory in amounts up to 0·05 per cent.

A. R. S.

419. Deodorising Liquid Naphtha Distillates and Mineral Oils. G. Petroff. E.P. 289,561. 1927.

The object of the process is the elimination of the unpleasant characteristic smell of light naphtha distillates by partial oxidation. The oil is charged into three inter-connected vessels. An oil-soluble catalyst such as manganese stearate is dissolved in the oil in the first vessel. The second vessel contains

copper turnings, and the third lead turnings. Air is introduced into the first vessel under pressure, and passes successively through the oil in each of the three vessels, carrying along certain volatile products with it which collect in a receiver provided. After 40 to 60 hours, the peculiar unpleasant smell is eliminated in all three vessels. The oil in the first vessel will require no further refining. That in the other vessels can be treated with acid and alkali in the usual way. H. S. G.

420. Refining Hydrocarbon Oils. P. McMichael. U.S.P. 1,669,944, 1928.

A gasoline can be refined without undue loss of unsaturated bodies to comply with the A.S.T.M. specifications for gasoline as regards colour, copper strip test, and doctor test by the following method: The unrefined gasoline is treated with an alkaline reducing agent, then an aqueous solution containing an oxide of a heavy metal and an alkali, after which it is treated with very dilute acidic solution. As the reducing agent sodium thiosulphate is suggested. This may be prepared for use by dissolving the thiosulphate to saturation in 30° Bé caustic soda. Alternatively, to insure that the solution is fresh and unoxidized for use, sodium bisulphite is dissolved in water and zinc dust added, after which the zinc compound is precipitated with milk of lime. Caustic soda is now added and the whole used as an emulsion.

The gasoline is agitated with this solution for 1 hour, water washed and subsequently agitated from 6–10 hours with a solution containing 10 per cent. of sodium chromate—copper sulphate in ammonia or litharge in soda may be alternatively used—and 1 per cent. caustic soda. The product thus obtained is sweet to doctor. The gasoline is then subjected to agitation with 1 per cent. hydrochloric, sulphuric or acetic acid, from 2–6 hours till the desired colour is obtained. A. R. S.

421. Method of Purifying and Decolorizing Petroleum Hydrocarbons. U.S.P. 1,674,020, 1928. Appl., 1-12-1924. A. Oberle and T. E. Scofield.

Light oils or distillates recovered by distillation either at atmospheric or increased pressure, or distillates recovered by vacuum distillation, are treated with an equeous solution of potassium iodide, the resulting mixture becoming either orange or pink in colour. The aqueous iodide solution is then drawn off and the distillate filtered, when its colour becomes considerably improved. After a final water wash, the resulting product is water white, and remains so even on exposure to direct sunlight. It is found that the iodide treatment must be carried out in sunlight, since light appears to have an activating influence on the reaction which occurs. H. E. T.

422. Treatment of Natural-Gas Gasoline, Casinghead Gasoline, Natural Gasoline and the Like. H. B. Bernard, assr. to Sinclair Oil and Gas Co. U.S.P. 1,658,322, 1928.

The invention provides an improved process of treating gasoline products derived from natural gas, casinghead gas and the like. The crude product is expanded into the upper end of a tower and made to flow in a substantially continuous stream through a helical path into a still, which is heated. The vapours from the still are taken upwardly through the tower but out of contact with the downward flowing stream of liquid. The treated gasoline products are withdrawn from the still and the separated vapours and gases from the top of the tower, which is cooled. S. W. C.

See also Abstract No. 440.

Kerosine.

423. Process for Treating Oils with Liquid Sulphur Dioxide. W. Hess, Assign. A.G. Chem. Ind. U.S.P. 1,674,676, 1928. Appl. 28-10-1926.

Hydrocarbon oils containing water are first treated with a minor quantity of liquid sulphur dioxide, and then separated from the sulphur dioxide extract, which contains the water.

The oil is then treated with a further, but larger quantity of liquid sulphur dioxide, in order to remove undesirable constituents. The sulphur dioxide is recovered from the first aqueous extract, and the second non-aqueous extract for re-use in the process. H. E. T.

See also Abstract No. 421.

Fuel Oil.

424. Process for Separating Hydrocarbons. C. B. Watson, Assr. to Pure Oil Co. U.S.P. 1,673,854, 1928. Appl. 26-12-1925.

A process is described for treating petroleum residues obtained from cracking or distillation plants. The residue is circulated through a pipe into a separating chamber, being forced in an atomized state into this chamber by introducing an inert gas under pressure into the conducting pipe. The temperature and turbulence of the residue in the chamber are maintained by introducing a further quantity of the heated inert gas. The lighter components of the residue escape through an outlet pipe at the top of the separating chamber to a condensing and collecting apparatus, while hot residual oil is continuously circulated through a closed ring, regular quantities of the hot oil being supplied to a number of burners situated adjacent to the ring. H. E. T.

See also Abstracts No. 432, 433.

Lubricating Oil and Wax.

425. Chemical Control of Grease Manufacture. C. B. Faught and F. R. Staley. *The Refiner*, 1928, 4, 72.

Cup greases, pressure lubricants and axle greases are usually made with lime, while sponge greases, fibre greases and gear greases are made with soda. The mineral oils used vary from light neutral to heavy cylinder stock. Axle greases are usually made cold from rosin oil and lime.

Fatty acids, which are by-products of the glycerine industry, are now widely used in the manufacture of greases. They have many advantages over the fats and vegetable oils, since they do not contain glycerine. The chief advantages are uniformity of composition; they yield a larger percentage of grease per unit of weight; they are more rapidly and completely saponified.

In making a cup grease, the standard practice is to put a weighed amount of fatty acid in the kettle with about half its weight of neutral oil. The oil and fat are melted with steam, the batch being agitated continually by paddles. When melted, the caustic solution or milk of lime is added. After saponification is complete, the procedure is continued until a comparatively dry brittle

texture is obtained. The water content of the soap is extremely important and requires careful control. After the soap is made, the neutral oil is added in a small stream over a period of several hours. The total quantity of oil added should be weighed in order to obtain the desired grade of grease. After all the oil is added, the grease is dropped through a filter into the filling kettle. Some manufacturers add a small quantity of perfume to cover the somewhat objectionable odour of calcium soap. This is added just before filling. Often the grease is coloured by means of aniline dye. This is an oil soluble dye and is dissolved in a small quantity of oil before adding it to the batch.

Rosin oil greases are made from rosin oil and lime. They are usually sold as axle greases, are made at atmospheric temperatures without heating, and consist of a lime soap and mineral oil, the latter being either a neutral oil or a black oil. They are comparatively simple to make.

If the raw materials and process are controlled by the laboratory, no difficulty is experienced in the manufacture, and a high-grade, uniform product can be maintained without waste. S. W. C.

426. Solubility of Lubricating Oil in Liquid Carbon Dioxide. E. L. Quinn. *Ind. and Eng. Chemistry*, 1928, **20**, 735–7.

The solubility of lubricating oil in liquid carbon dioxide has been determined at various temperatures, ranging from $-20°$ C. to $+25°$ C. The maximum solubility was found to occur at a temperature of 10° C., at which temperature the solution contained 0·9 per cent. of oil.

From density determinations it was found that lubricating oil had little effect on the density of liquid carbon dioxide at ordinary room temperatures.

The solubility of glycerol in liquid carbon dioxide was found to be very small, in all probability less than 0·05 per cent.

The maximum solubility of the oil occurred at that temperature, at which the densities of the two components are equal. H. E. T.

427. Improvements in or Relating to Mineral Lubricating and Transformer Oils Exposed at Raised Temperatures to Air. F. Hoffman and I. M. Dunkel. E.P. 262,107, 1928.

Mineral transformer and lubricating oils are prevented from oxidation with air or oxygen at higher temperatures by the addition of small quantities of aldehydes, and their products of polymerization with either ammonia or organic amines.

It is claimed that in this way the oxidation of the oil is substantially reduced. A. R. S.

428. Improvements in the Manufacture of Refined Lubricating and Like Oils. De Bataaf. Petr. Maatsch. E.P. 263,167, 1928.

The invention provides a process for the manufacture of refined lubricating and other oils having a low congealing point or a low cloud test from raw materials containing paraffin. The raw material is first separated into an extract and a "refined product" containing paraffin by treatment with liquid sulphur dioxide. The paraffin-containing product, whilst saturated with sulphurous acid, is then filtered at a low temperature (0° C. or lower) either by means of the vapour pressure of the sulphurous acid or a pump and the sulphur dioxide finally evaporated off. The de-paraffined "refined product" and the "extract" when combined produce an oil having a low set point and a low cloud test, whilst the viscosity is practically the same as that of the raw material. S. W. C.

429. A Process for the Distillation of Oils Occurring in the Petroleum, Tar and Similar Industries, More Particularly for the Production of Lubricants. L. Steinschneider. E.P. 264,476, 1928.

The invention provides apparatus for producing lubricating oils from suitable stock comprising a tube still through which the liquid material is forced, an evaporator in which the decomposed products and preferably the lighter constituents are removed from the heated stock, further distilling apparatus in which the desired lubricating oil is taken off from the liquid material, passing the evaporator, means for producing different pressures in the distilling apparatus or for producing different partial pressures in the oil vapours of the distilling chambers, means for cooling and condensing the desired lubricating oil and the decomposed products independently from each other and preferably means for circulating the liquid material between the tube still and the distilling chambers. The apparatus may comprise a set of such units in which the liquid is heated up to the final temperature step by step by passing the units one after the other. The distilling chamber in which the decomposed products are removed is held under a slight vacuum, and that in which the desired hydrocarbons are taken off under a much higher vacuum. S. W. C.

430. Improved Method and Apparatus for Refining Oils. Simplex Refining Co. E.P. 283,383, 1928.

The invention relates to the art of oil refining and particularly to means of raising oil from one level to another, and it has a peculiar utility in lubricating oil refining plants operating under high vacuum. The handling of oils in vacuum plants by mechanical pumps presents certain difficulties, especially as it is extremely difficult to keep packed joints tight when pumping liquid at the high temperatures used in such plants. In lubricating oil plants operating under high vacuum, any such leakage is especially objectionable, since it results in the vacuum pulling air into the system and even small amounts of air leaking into such a system not only combine with the oil to produce objectionable compounds in the oil, but also produce gaseous products which have a large volume due to the low absolute pressure.

The invention may be illustrated by its application to a vacuum lubricating oil refining plant. Oil vapours are passed into the base of a dephlegmator which contains a number of plates, over which cooled oil is passed. The condensed oil collects in the bottom of the dephlegmator and passes into a circulation cooler, from the bottom of which a pipe leads down, turns in a U-bend and enters the top of the dephlegmator. The arm of the U-bend which leads to the top of the dephlegmator is perforated for a short distance at its lower end and jacketed. Means are provided for passing steam into the jacket. Oil will collect in the circulation cooler and U-bend and a static head will be provided. If now steam be passed into the jacket it will enter the pipe, mix with the oil and so lighten the oil in that limb, that the static head produced by the oil in the circulation cooler will be sufficient to force the oil-steam mixture up the pipe and into the top of the dephlegmator. Here the oil passes downward over the plates, condensing the up-flowing oil vapours and the steam is set free and taken out of the plant by means of a steam ejector which passes it into a chamber held at a pressure of about 60 millimetres by means of a pump. The steam can be readily condensed at this pressure, whereas at the pressure in the plant—20 millimetres—considerable difficulty would be experienced. Condensed oil is continually withdrawn from the circulation cooler by means of a pump.

Steam for lifting the oil is merely used as a convenience, it being possible to use anything which is a gas at the temperature and pressure of the plant and which does not react with the oil. S. W. C.

431. Purification of Petroleum Oils for Removal of Sulphonated Compounds. R. C. Moran, Assr. to Vacuum Oil Co. U.S.P. 1,659,782, 1928.

The invention provides a process of preparing a refined hydrocarbon oil which consists in subjecting a lubricating oil fraction to successive treatment with fuming sulphuric acid, removing the treated oil, admixing therewith 8 per cent. of ethylene glycol containing less than 1 per cent. of water or a solution of ethylene glycol in ethyl alcohol, etc., either with or without previously washing the oil with alkali, and subsequently separating the ethylene glycol therefrom, thereby removing sulphonated compounds from the oil. S. W. C.

432. Process of Refining Mineral Oil. H. T. Maitland, Assr. to Sun Oil Co. U.S.P. 1,668,920, 1928. Appl. 21-12-1921.

The author describes a method of refining mineral oils and heavy distillates such as lubricating oil stock. Sand run through a forty-mesh screen and caught on a sixty-mesh screen is coated with sulphuric acid, 20 parts of acid being used to every 500 parts of sand. The oil stock is then run slowly through the sand. The efficiency of the acid-coated sand only becomes impaired after at least seven thousand parts by volume of the oil have run through it. The outflowing oil contains a very small proportion of acid, and practically no resins, the latter remaining in the sand bed. To obtain an oil of the best quality, the acid carried through by the oil is promptly neutralised by alkali, or the oil is filtered through fullers earth.

In place of sand, such substances as glass beads, spent earth, particles of brick or clay may be used. An essential property of the base used, however, is that it must be non-absorbent and must also present a large superficial area.

It is claimed that the quantity of sulphuric acid used in this process can be as low as ¼ per cent., whereas in the ordinary acid alkali process, the quantity of acid used is usually 4 per cent. by volume of the oil treated. H. E. T.

433. Art of Refining Oils. C. R. Wagner. U.S.P. 1,669,151. Appl. 17-7-1922.

This invention deals with the separation of "petrolatum" or amorphous wax from steam reduced crude oils, cylinder stocks or similar oils. Effective separation may be produced by diluting the stock with an oil solvent, in which wax is materially less soluble than in naphtha, chilling the mixture to a suitable temperature, and separating the wax by settling. When butanone or methylethylketone is employed as a diluent, a better separation of wax and a wax-free oil with a lower cold test is obtained, than when naphtha is employed. On standing, the wax present separates out and is then removed. The diluted oil may be subjected to a steam distillation, in order to recover the butanone, which can be again used. Methylethylketone which has not been perfectly dehydrated further decreases the solubility of the petrolatum. H. E. T.

434. Art of Refining Oils. C. R. Wagner. U.S.P. 1,669,180, 1928. Divided. Appl. 31-5-1924.

This invention is an extension of U.S.P. 1,669,151. As diluents, mixtures of normal or secondary butyl alcohol and naphtha, or of methylethylketone

and naphtha may be employed. A mixture of 25 per cent. methylethylketone and 75 per cent. naphtha has a solubility for petroleum of less than 75 per cent. of that of naphtha, while a mixture of 50 per cent. methylethylketone and 50 per cent. naphtha has a solubility for petroleum of approximately 30 per cent. that of naphtha. H. E. T.

435. Art of Refining Oils and Other Liquids. O. Werner. U.S.P. 1,669,182, 1928. Appl. Nov. 10, 1925.

A method and apparatus are described for recovering used lubricating oils by removing the lighter constituents such as gasoline, which have mixed with the lubricating oil during use in internal combustion engines. The light constituents of the oil are driven off by utilising mercury vapours as a source of heat, when the risk of fire is decreased and high temperatures are obtained at low pressures. The heat applied to the oil is automatically controlled and maintained at any desired temperature without danger of local overheating or excessive carbon formation.

In practice, a working temperature of 600° F. for the mercury vapour, which heats the oil, can be obtained at the comparatively low pressure of 5 lbs. per square inch. This invention may be quite readily adapted for various uses with different kinds of oils to be distilled and refined.

H. E. T.

436. Improved Method and Apparatus for the Regeneration of Lubricating Oils which have been Contaminated in Thermal Engines. L. Mirlesse and A. I. Kousnetzoff. E.P. 269,148, 1928.

The process provides a method of regenerating lubricating oils contaminated in thermal engines consisting in filtering the oil, whilst cold in a suitable filter, interposed in the supply pipe, and from which the oil is passed through a heating chamber in a thin wide stream, the heating chamber being provided with internal horizontal walls arranged in staggered relation, branch pipes leading from the exhaust pipe of the engine being passed through the chamber and lagging being placed around the outside. The water vapours and light hydrocarbons produced in the heating chamber are discharged through a pipe which leads either to the atmosphere or to the float chamber of a carburettor or to the induction pipe of the engine, or to a feed tank for the liquid fuel for the engine. The oil leaving the heating chamber is again filtered and thus regenerated and subsequently passed to the distributing passages of the engine. S. W. C.

Special Products.

437. Special Naphthas with Pipe Still. C. O. Willson. *Oil and Gas J.*, 1928, **27**, No. 5, 154.

The Primrose Refining Company has turned its attention to the manufacture of three special grades of naphtha, utilising for the purpose a pipe still in co-operation with some old shell stills. The naphthas referred to are rubber solvents, V.M.P. naphtha and cleaner's naphtha, these products being so chosen as to cover the range of light end-products previously obtained. The cleaner's naphtha is a special cut known as a Stoddard solvent, 50 per cent. being the maximum volatile below 350° F. (177° C.) and the end-point not exceeding 410° F. (210° C.). The rubber solvents usually have an initial boiling point below 52° C. and an end-point not greater than 152° C. The V.M.P.

naphthas are used as turpentine substitutes and their specifications vary somewhat widely. Archer County fields crude (A.P.I. gravity 40) after preheating is charged into the middle of a baffle tower superimposed on a shell still, the tower being 3 by 15 feet and the still 9 by 20 feet. The overhead enters a second 3 by 10 foot tower partially filled with crushed granite, 15 per cent. of the crude being taken off as rubber solvent. The overhead stream is controlled by spraying reflux into the top of the column, the whole of the run back falling into the still, the contents of which are then charged to the pipe still. The latter consists of 1800 feet of tubing, the roof tubes being 3 inch O.D. and the main bank of tubes 4 inches O.D. The oil leaves the still at 700° F. (370° C.) and enters an evaporator at the base of a 5 by 68 foot tower divided into two sections, the upper containing 14 and the lower 10 bubble decks. The overhead constitutes V.M.P. naphtha, the draw-off between the two sections being Stoddard solvent, and that from the base of the lower section a mixture of kerosine and gas oil. Refluxing for the V.M.P. naphtha takes place in the top of the tower, utilising an open stream of the same specification as is taken overhead. The same type control is used for the Stoddard solvent, the reflux of the required specification entering the tower at the deck, which is just below the upper section. A steam coil is also used at the base of the upper section of the tower. The stream of gas oil and kerosine flows to a second shell still, the latter being taken overhead. About 80 per cent. of the crude is vaporized, 38 per cent. being refined into special naphthas and 14 per cent. into kerosine.

Note.—There is some confusion in the article between V.M.P. naphthas and Stoddard solvent. H. G. S.

438. Art of Making Asphalt. E. A. Rudigier. U.S.P. 1,673,533, 1928.

Heavy asphaltic residue is introduced into a large tank fitted round its base with a pipe for the supply of air. The tank itself is heavily lagged and an exit tube for air and vapours, which is connected to a tower filled with brick checkerwork, down which water flows, is fitted to the top of the tank.

The residuum oil is introduced at a temperature of 420–440° F., and air, preferably pre-heated, is introduced at 11–12 lbs. gauge pressure, delivering for about 40 hours, at 900–1000 cubic feet in a tank 25 feet in diameter by 30 feet high.

The heat of oxidation keeps the temperature up during the run and the hydrocarbon vapours given off are condensed in the tower and run to a catch-pot. A. R. S.

See also Abstracts 400, 401.

Analysis and Testing.

439. A Micro Saybolt Type Viscometer. S. M. Rogers and L. R. Adkins. *Ind. and Eng. Chem.*, 1928, 20, VII., 742.

The viscometer is made of metal, can be made to fit any usual type of bath and uses as little as 8·5 c.cs. of sample, the actual capacity being 8·4 c.cs., while the capacity including overflow is 12 c.cs.

The dimensions of the barrel are 6·7 cms. high and 1·43 cm. diameter and the standard 60 c.cs. graduated receiver is replaced by a 10 c.cs. cylinder, 7 c.cs. being run out.

Check testing is well within the tolerance for the Saybolt universal viscometer. A. R. S.

440. THE ESTIMATION OF WATER IN BENZOL SPIRIT MIXTURES. D. Peters. *Petr. Zeitschr.*, 1928, **24**, 741–743.

This is important in the production of absolute alcohol, which is almost always effected by distillation with benzol by Young's process, and also in the preparation of motor fuels.

Young's method of separating benzol and spirit by washing with water and fractionating the alcohol from the spirit is unsuitable as it takes too much time. Crismer has shown that every high per cent. alcohol, when mixed with an equal volume of petroleum, exhibits a peculiar critical solution temperature depending on its water content. He determined the critical solution temperatures of mixtures of various petroleums with different high per cent. alcohols, and tabulated these, and by this means was able to determine the percentage content of an unknown alcohol. The process was as follows: The requisite quantity of benzol was added to pure alcohol and both liquids were united by means of burettes. This mixture, with known benzol content, was added to the same volume of petroleum and the de-mixing temperatures determined. These were represented on a graph with the ordinates as temperatures and abscissae as the benzol content. The de-mixing temperatures for mixtures of such alcohols with different quantities of benzol were found to lie on an approximately straight line, the increase of benzol content corresponding with decrease of de-mixing temperatures. The benzol also acted as a solvent for spirit and petroleum. In this way the de-mixing temperatures for a whole series of various per cent. alcohols with increasing benzol content were established and found to lie for the different per cent. alcohols on approximately parallel lines. The distance between these parallels was smaller the more water the alcohol contained, and naturally the de-mixing temperatures were generally higher the lower the per cent. alcohol.

This method depended on the boiling point of the mixture utilised—i.e., about 70°. On the other hand, the determinations with greater benzol content were inaccurate; for example, if the alcohol was greater than 99·4 per cent. pure, then the determination with a benzol content of greater than 30 per cent. was inaccurate. A sharp bend occurred in the curve for 98·5–99·7 per cent. alcohol with 50–60 per cent. (by volume) of benzol. A similar phenomenon occurred with benzol-benzene spirit mixtures. The curve of 95·7 per cent. alcohol exhibited with 13–15 per cent. by volume of benzol and 55–64 per cent. benzol, peculiar deviations similar to that shown with 96·4 per cent. alcohol with 13–14 per cent. benzol. In this way the de-mixing temperatures with different water and benzol contents were determined and shown graphically. The geometric locus of these de-mixing temperatures was represented by a number of lines by which means the degree of the alcohol could be determined in any spirit-benzol quantity. The benzol content of the quantity had first to be estimated by shaking with cold water (12° C.), since benzol is not very soluble in strongly diluted spirit; 2 c.cs. of the mixture were treated with 2 c.cs. petroleum from a burette. The de-mixing temperature was determined by slow cooling, using a thermometer marked to tenths of a degree. When the mixture had separated at room temperature, it was heated until it had dissolved in an Ehrlenmeyer flask in a water bath, which was very slowly heated. The mixture was then left in the bath and stirred with the thermometer and allowed to cool very slowly until the mixture separated.

The alcohol exhibiting the found de-mixing temperature, with the definite benzol content, was then sought from the table, thus giving the percentage content of alcohol mixed with benzol. The accuracy of this determination amounted to $\pm$0·05 per cent. water content. The petroleum used had a density D^{15}/ 0·808. W. S. C.

Refinery Plant.

441. Operating a Continuous Plant for Refining Distillates. J. C. Morrell and D. J. Bergman. *Chem. Met. Eng.*, 1928, **35**, 350–4.

A method of operating a continuous treating plant for refining distillates is described. The design, dimensions and characteristics of the acid mixing, plumbite and caustic orifice columns and settling tanks are given. The system, which is continuous, consists of a combination of mixing, settling and washing apparatus, the order depending on the method of treatment. The pressure distillate and acid are pumped through a mechanical mixer, the distillate and acid sludge thence flowing into a settling tank where sufficient time is given for the acid sludge to settle. The overflow goes to a washing tank, where the oil is sprayed with water to remove acid still present. The oil is next passed into a plumbite mixer and thence to a plumbite settling tank. Without any further washing, the oil is now transferred to a storage tank in which some caustic settles out. It has been found that the deposited caustic protects the bottom of the tank from corrosion and imparts a sweeter odour to the pressure distillate on distillation. The gasoline recovered by distillation is given a final weak soda wash. H. E. T.

442. Modernisation of Cylindrical Stills. C. W. Stratford. *Oil and Gas. J.*, 1928, 26, No. 43, 164.

This paper gives an account of some results obtained by the Associated Oil Co. as a result of experimental work on the improvement of distillation methods with a view to reducing refining costs. The work dealt with concerns the firing of stills and the circulation of oil within. The experimenters worked to improve existing shell stills rather than turn to the then existing and more efficient pipe stills for two reasons: the price of pipe stills was high and their company considered it inadvisable to discard their plant of shell stills in which a large amount of capital was sunk.

The oil in the still is circulated by means of a centrifugal pump, the delivery being directed in high velocity jets against the still surfaces exposed to heating. The oil spreads over the surface in a rapidly moving sheet, both keeping the surface clean and acting in the most efficient way to take up heat from it. A forced draught fan feeds air to the furnace and supplies the energy required for intimate mixing of air and oil fuel and also to keep the furnace gases spinning and sweeping over the still surfaces.

The first four stills equipped were 10 feet in diameter by 40 feet long. The circulating pump delivered 750 gallons per minute of oil against the still bottom. The stills were worked continuously vaporizing about 42 per cent. of crude and a charging capacity of 5300 barrels was attained. The furnace air was used to cool the furnace walls and entered the wind box at about 300° F.

Six more stills 12 feet by 45 feet were adapted to the system with a circulation of 1100 G.P.M. These stills were run at 6500 barrels per day, each with 40 to 50 per cent. evaporation. A short test of maximum capacity reached a rate equal to 9400 barrels per day on one still.

One other 12 foot by 45 foot still was fitted with a pump delivering 3600 G.P.M. at 35 lbs. per square inch and 9200 barrels per day was reached. A bank of tubes was added to this still to extract heat from the flue gases, average flue gas temperature through the bank falling from 1200° F. to 585°

or 700° F., the latter temperature depending on temperature of oil in the tubes. Tubes were also fixed along the front face of the bridge wall to take up heat by radiation and prevent overheating of the wall. These tubes proved unnecessary, but provided a possibly useful source of heat in certain special distillation flow arrangements.

All the results just quoted are on the low side, due to inadequate flue capacity, and the writer estimates the probable capacity of a 12 foot by 45 foot still, running refined crude and vaporizing 50 per cent., at between 10,000 and 12,000 barrels a day, the still being fitted with a flue gas economiser.

Results of operation of oil circulation stills show marked saving in refining costs. The first effect of applying the stills to fractionating equipment and running on refining crude was to close down a number of shell stills and pipe still toppers. Re-run stills were eliminated by the use of direct fractionation. With circulation of oil the use of steam for agitation was unnecessary and several boilers were closed down. Overall fuel consumption has been reduced to less than half the previous quantity and now varies mainly between 0·9 per cent. and 1·3 per cent. by volume.

The writer deals with certain ideas realised during the experiments and in particular with the application of fire tube heating to circulating stills. Although in certain existing fire tube stills coking is experienced if the combustion is actually allowed to take place in the tube instead of in a preliminary oven, he contends that with efficient forced circulation this should not occur.

The following are his proposals for converting a 10 foot by 30 foot still. A single fire tube about 3½ feet in diameter would supply the necessary heating surface and around it would be wound the oil distributing manifold with jets directed at the tube. A mechanical atomising burner could be applied directly to the tube and arranged to spin the gases at such a rate that they are moving over the surface at over 100 feet per second for the whole length. At the outlet from the furnace tube some of the spinning energy could be converted to pressure by "unwinding" the gases through a volute before passing them on to either an air pre-heater or other form of economiser.

Two forms of "direct heater" are described, the first being in the form of a small diameter tube wound on a cylindrical refractor former and surrounded by an annular firing space and a hollow cylindrical furnace wall. The oil moving at high velocity through the spiral is flung by centrifugal force against the outer, or heated, half of the tube section, while any gases liberated rise to the inner side of the tube where heating is less intense. Several disadvantages of this type are quoted and the writer proceeds to deal with another form of direct heater. This heater, which includes the oil circulation system, consists of a firing tube against the outside of which the oil jets impinge, the firing tube being surrounded by another tube allowing a relatively small annular space for the oil. Two manifolds surround this jacket, one for supplying the circulating oil under pressure to the jets, and the other for collecting the hot oil and delivering it direct to the vaporiser. The circulating pump draws oil from the vaporiser and delivers it together with some fresh feed oil to the jet manifold. Charging rate would affect the time factor, but not the velocity of oil over the heated surface, and the heater has the advantage of being able to be opened for inspection.

The importance of using an efficient oil burner is dealt with and also the advantage of causing the hot gases to move at high velocity over the still plates. Many difficulties encountered when firing strongly are put down to too low a liquid velocity over heated surfaces. E. R. B. S.

Engineering.

443. Some Considerations regarding the Peak-Load Problem and High-Powered Peak-Load Diesel Engines. M. Gercke. Diesel Engine Users' Association. May 1, 1928.

After dealing with the general question of peak-load problems in electricity supply the author discusses the comparison of primary peak-load units. The main advantage of the high-powered Diesel is its ease of starting without any preliminaries, the time from starting to full load being 3 to 6 minutes in the case of the largest Diesel units in existence. In addition to the vertical four-stroke Diesel with airless injection for outputs up to 15,000 kw., the single- and double-acting two-stroke types are being increasingly used for peak-load purposes. The largest units in use or under construction are :—

Slow-Speed types.—Two-stroke double-acting 9-cyl. unit of 1500 mm. stroke, 860 mm. diam. Speed, 94 r.p.m. Output, 15,000 h.p., or about 10,500 kw.

High-Speed types.—Two-stroke double-acting 10-cyl. unit of 900 mm. stroke, 600 mm. diam. Speed, 215 r.p.m. Output, 11,700 h.p., or about 8000 kw. gross, 7500 kw. net. Airless injection.

The initial cost of these units, installed in Germany, was about £16 10s. per kw. for the slow-speed and £11 per kw. for the high-speed, including electrical equipment and building, but excluding the site. Average fuel consumption is from 0·55 to 0·575 lb per effective kw.-hour. Comparison of operating costs of steam power plants and of Diesel engines shows that peak-load generating costs are practically equal. Peaks have an unfavourable effect on steam plants, the Diesel being the best engine to cut off peaks and to generate peak units. Diesel peak-load installations should be as simple and as fool-proof as possible, and should include, in addition to the main Diesel sets, compressed air plant, fuel tanks, pumps, etc., lubricating oil tanks, cooling water plant, scavenging air plant, exhaust through silencers and exhaust tanks, positive starting devices and arrangements for practically silent service in residential districts. Tests with the loop method of scavenging have proved that it can be safely used at high speeds with good results.

G. S.

444. Process and Apparatus for Producing a Combustible Charge for Use in an Internal-Combustion Engine. D. Balachowsky and P. Caire. E.P. 265,186, 10-1-1927. Conv. date (France), 27-1-1926.

The process consists in atomising the hydrocarbon with a volume of air not exceeding one-half of the air required for combustion. The rich mixture is then divided as completely as possible by causing it to pass through a nest of tubes heated by exhaust gases to 300–350° C., and is then passed to the engine through a common outlet provided with an additional air inlet device for adding supplementary air in sufficient quantity to produce a combustible mixture. The nest of tubes is made of one of the known metals which, when heated, effects catalytic decomposition of hydrocarbons. Each tube contains a rod of one of the same metals twisted or formed so as to give a whirling motion to the rich mixture. In any part of the tubes may be positioned a porous mass, preferably gauze or filings of copper, for the purpose of retaining the surplus non-vaporized liquid globules in order to promote subsequent vaporization and catalysis.

H. S. G.

445. Improvements in the Operation of Internal-Combustion Engines. I. G. Farbenind. A. G. B.P. 274,462, 1928.

This patent covers the use, in internal-combustion engines, of a pulverised fuel consisting of a mixture of mineral coal and lignite. As an example,

good results are stated to have been obtained from a mixture of coal dust and lignite dust containing 80 per cent. of mineral coal by weight.

E. R. B. S.

Anti-Detonation.

446. Flame Movement in Gaseous Explosive Mixtures. O. C. de C. Ellis. *Fuel*, 1928, VII., 245–252.

Part II. Propagation of Flame within a Soap Bubble.—In studying explosions within a soap bubble it was early observed that the position of the point of ignition mattered little as the soap film is broken long before the flame reaches it, so that only the gas immediately surrounding the spark is ever ignited. It was observed that the spread of flame with a weak mixture of carbon monoxide and oxygen is not equally fast in all directions, a lifting pressure flattening the flame at its base and eventually developing it into a cusp.

Part III. Propagation of Flame from Central Ignition in a Sphere of Glass.—Spheres of different sizes were used according to the strength of the explosion to be developed. With strongly explosive mixtures it was found that the flame spreads equally fast in all directions, but with weakly explosive mixtures portions at the bottom of the sphere are burnt last. A 46 per cent. CO mixture with air was found to have the maximum explosion speed of CO-air mixtures; similarly, a 7.5 per cent. CS_2 mixture with air burnt the most rapidly of such mixtures. It was shown that a series of rapidly successive photographs of the spread of flame gives a more accurate conception of the behaviours of the flame than the record obtained by a rotating film continually exposed to the flame. A series of photographs illustrates the spread of flame in a weakly explosive mixture ignited near the base of the sphere, the unburnt layer at the bottom of the sphere forcing the under portion of the flame front into a cusp: with very weak mixtures the flame expires before it reaches the bottom of the vessel.

W. N. H.

447. Flame Movement in Gaseous Explosive Mixtures. O. C. de C. Ellis. *Fuel*, 1928, VII., 300–4.

Part IV. Propagation of Flame from Eccentric Ignition within a Sphere.—Superimposed successive photographs are shown of weakly explosive mixtures ignited eccentrically, the combined upthrust and centripetence of the flame producing interesting movements. It is shown that central ignition gives the greatest rise in pressure in an exploded sphere of gas compared with the pressures obtained with any other ignition position, this being due to the cooling effect of the walls. Ignition at the top of the sphere gives the smallest rise in pressure as the flame produced by that ignition has a maximum area in contact with the walls during its spread.

W. N. H.

448. A New Way of Checking Detonation. *Petr. Times*, 1928, 1,006.

Dr. Sokal of the Kalamite Corporation of Brooklyn coats the cylinder head and exhaust valves of an internal combustion engine first with a silicate binder and then, when dry, with a mixture of litharge and binder. The litharge changes during ordinary running into lead carbonate. At the explosion of the mixture in the cylinder, however, the temperature, particularly if the engine is detonating, is sufficiently high to convert the carbonate into litharge. This conversion, being an endothermic reaction, lowers the temperature of the walls of the combustion spaces, thus leading to cooler running in general and eliminating detonation. Road tests on a Chrysler car fitted with a special high-compression head tended to confirm the above claim. It is further

claimed that the coating reduces carbon deposition, but in any case, the coating remains effective under any reasonably porous layer of carbon.

W. N. H.

See also Abstract No. 405.

Cracking.

449. Vapour Phase Cracking in Refining Oil. (The Knox Process.) D. M. Evans, Petroleum Conversion Co. *Oil and Gas J.*, July 12, 1928, p. 146.

The author discusses the advantages and disadvantages of vapour phase cracking. The disadvantages do not apply to vapour phase cracking alone but to a certain extent to every process wherein heat is applied through metal walls, for no metal commercially applicable can withstand the prolonged high temperatures necessary to crack petroleum without rapid deterioration and lowering of tensile strength. This results in operating at reduced temperatures and pressures, thus reducing capacity, or periodic renewal of costly equipment, or working at an enormous hazard to life and property.

As long as oil is confined within a metal container heated externally, that portion of the oil nearest the walls of the vessel or tube will be overheated and overcracked before the inner portion of the oil attains cracking temperature. Realising these facts, W. J. Knox conducted a series of experiments which resulted in the patenting of the Knox Process wherein the principle of direct internal heating is applied. The process has been recently improved and put in operation on a commercial scale, and the following advantages are claimed:

(1) It produces unequalled antiknock motor spirit.

(2) It applies direct internal heat, resulting in an evenly cracked product without excessive formation of gas and coke.

(3) It uses standard blast furnace stoves as heat generators, assuring utmost fuel economy.

(4) It has a novel method of continuous carbon removal.

(5) All operations above 400° C. are conducted in metal vessels lined with refractory and insulating brick to reduce radiation losses and prevent corrosion or overheating of the metal.

(6) All fractionation is carried out at low temperatures, the final temperature of the rectifying column being not more than 95° C. (200° F.).

(7) Charging stock containing water in any proportion may be used without danger.

(8) Units with a capacity of 2,000 to 4,000 brls. per day may be constructed with perfect safety, for the process is not hampered by high pressure or high temperature.

(9) Oils with a high sulphur content may be cracked without additional cost.

(10) Three men can operate a 2,000 brl. gasoline per day unit thus reducing labour costs.

The process is arranged as a closed cycle. Non-condensable gas serves as the heat carrier, being heated continuously by passage through the heated brick chequer work of hot blast stoves. Four of these stoves form the heating section of a 2,000 brl. unit, and are operated so that each is fired for three hours, and furnishes heat to the carrier gases for an hour. The standard stove is a vertical chamber with a combustion chamber at the bottom and a dome at top. The vessel is lined with refractory material and a chequer work of firebrick is built within, leaving a central flue. While heating the chamber, the hot gases from the combustion chamber travel upward through the central flue into the dome, then downwards through the firebrick filling, thus

storing a large quantity of heat. At the end of the firing period the top temperature is about 1,000° C. At this temperature the firing is stopped, the stove purged of the products of combustion or air, and gas from the cracking process introduced, then switched into the cracking system of the plant whilst the spent stove is cut out for further heating.

The circulation of the carrier gas is the reverse of the flue gas circulation during the firing period, and emerges from the stove at about 900° C. This temperature is reduced to about 750° C. by low temperature gas from the heat exchangers before entering the reaction zone, where it comes in contact with oil vapours from the vaporiser in a specially-designed mixer. Sufficient quantities of carrier gas and oil vapour are introduced to maintain a temperature of about 540° C. Heat of reaction and radiation loss reduce this to about 480° C. at the outlet of the cracking zone. The average temperature with 4 minutes' reaction time is sufficient to yield 25 per cent. gasoline. Leaving the reaction zone, the gases go through a scrubber column and pass upward counter current to a spray of oil. Any carbon is washed from the gas stream and is blown down as fuel whilst excess heat is absorbed and returned by heat exchange to the carrier gas. The carrier gas and vapours enter the condenser wherein the gasoline drops out, then the gas is slightly compressed and passed through an absorption system where the remaining gasoline is recovered.

The stripped gas enters the scrubber oil heat exchangers and is raised from 40° C. to 300° C. approximately, and at this temperature enters the bottom chequer work of the hot stove, thus completing the cycle. R. P.

450. Catalysts in Oil Cracking. *Petr. Times*, June 2, 1928, p. 1.020.

Many attempts to accelerate cracking by the use of catalysts have been made. In a recent issue of *Die Metallborse*, H. Pincass gives particulars of certain cracking processes in which catalysts are used. Aluminium chloride is known to act on the heavy hydrocarbons of petroleum, producing high yields of light hydrocarbons. According to American patent 1,193,540 this reagent is used at a temperature sufficient to vaporize the oil, and yields a product whose boiling range is not greatly over that of naphtha.

Another American patent, 1,476,091, specifies a still provided with agitators or metal balls which give a large contact surface for the reagent and the hydrocarbons. Other patents concerned with this reaction are American 1,478,438, 1,473,316, 1,476,219, and British patent 1,931,188. The quantity of aluminium chloride used varies from 5 per cent. to 10 per cent. and temperatures range from 200° C to 800° C. The manufacture and recovery of anhydrous aluminium chloride is a problem in itself. American patent 1,157,802 describes its manufacture from bauxite and chlorine, whilst another, 1,544,328, proposes to make the catalyst from aluminium hydrate, active carbon and chlorine. A Swiss patent, 111,354, describes the preparation of a special catalyst composed of aluminium chloride, an alkali or alkaline earth, a silicate, and a mineral oil high in sulphur and unsaturated hydrocarbons. This forms a pitch-like mass, which is not hygroscopic.

The regeneration of aluminium chloride is accomplished by heating the used material with oil and condensing the vapours, according to a whole series of patents.

It is doubtful if aluminium chloride can be regarded as a true catalyst as relatively large quantities of the substance are required.

Catalysts other than aluminium chloride such as active carbon, hydrosilicates, such as Florida and Georgia earths, but not ordinary Fuller's earth or clays, may be used. The hydrosilicate removes and retains asphaltic substances from the oil. After removal of light fractions the residue may

be used directly as lubricating oil without acid treatment. The quantity used varies from 5 to 25 per cent.

Nickel Carbonyl, Swiss Patent 114,228, is said to be very effective as an accelerator in cracking of oils.

The use of oxides of the rare earths is covered by a series of patents. Mercury vapour in the presence of hydrogen either under pressure or vacuum is claimed to give a high yield of gasoline which requires no further refining. (U.S. pat. 1,423,709). Metals including iron, copper, zinc and their compounds are proposed, but it is doubtful whether they act as effectively in cracking as in the synthesis of ammonia. Mixtures such as zinc chloride, magnesium chloride and burnt lime, or the sulphides of iron and antimony, potassium carbonate, and calcium chloride are also proposed as catalysts.

Metal shavings of copper, iron and aluminium as well as titanium oxide, iron-copper, nickel oxide and magnesia are reputed to be effective.

These brief references to efforts in cracking with catalysts give an indication of the directions in which the problem is being attacked, and it seems reasonable to anticipate some valuable results from the activity. R. P.

451. Cracking Survey. *Refiner*, 1928, No. 6, 70.

A recent survey of liquid phase cracking installations indicates that although some millions of dollars are being spent in the erection of new units, developments are not so extensive as during the boom period of three years ago. Tables are given showing the locations and capacities of various plants erected during the last six months, and installations now under construction. In the former category, 25 units ranging in capacity from 1,000 to 3,000 brls. daily, and having a total daily capacity of 53,100 brls. are noted. Of these, six were laid down in countries outside the U.S.A., their total capacity being 12,200 brls. One Cross unit was built in Patagonia (1,000 brls.) and Dubbs units in Canada (1 unit), Dutch East Indies (2 units), and Rumania (2 units). Installations now under construction total 34 (total capacity 60,700 brls.), of which 16 are being erected in U.S.A. A second Cross unit is under erection in Patagonia, two 2,000-brl. Tube and Tank process at Fawley, England, and a 1,000-brl. unit at Bahia Blanca, whilst a Holmes-Manley plant is under construction at Fiume, Italy. Dubbs cracking plants are being installed at Balik Papan, Borneo (six 1,000-brl. units), in Java (one 1,000-brl. units) and at Curacao (four 1,000-brl. units). The Burma Oil Company is also building one 1,500-brl. Dubbs plant at Rangoon and a 1,000-brl. unit is being installed by the Aquila Franco Romana at Ploesti. Details of new plants under construction in U.S.A. are also given, the tendency being to adopt larger units of greater through-put capacity. H. G. S.

452. Treating Oils to Promote Cracking. M. B. Schuster. E.P. 289,556. 31-1-1927.

A process for increasing the yield of volatile products by cracking, characterised by incorporating in the material to be treated a colloidal cracking agent, such as bentonite or certain kinds of colloidal hydrated lime, and then distilling under pressure. The colloidal material may be added dry or first dispersed into the form of a gel by mixing with a hydrocarbon, water or alcohol. The residue from the process is closely allied to, and is an effective substitute for, natural asphalt. H. S. G.

453. Art of Heat Conversion of Hydrocarbon Oils. F. A. Howard and N. E. Loomis. U.S.P. 1,670,037, 1928.

In this process oil is pumped through a preheater into a coil wherein the temperature of the oil is raised to about 450° C. The stock is fed through

this coil at such a rate that substantially no coke is formed, a suitable rate being about 2,000 gallons per hour for a coil 4 in. diameter and about 1,600 feet long. After passing from the coil, the oil enters at the bottom of the first drum of a series. In the form of apparatus shown two drums or enlarged chambers are used. These drums are of such a size that the oil remains in them for a considerable period, *e.g.*, 1½ to 2 hours. The first drum is lagged with heat insulating material and the oil may drop 50° in temperature. A considerable amount of coke is deposited in this drum, but is readily removable and a substantial conversion into low boiling products takes place. The oil from this drum leaves at the top and enters the second of the series. This drum is mounted over a furnace so that it may be mildly heated, and produce a greater yield of gasoline.

Alternatively, the second drum may be lagged and the necessary amount of heat supplied internally by introducing air in regulated quantity. At the temperatures prevailing within the drum, the supply of oxygen causes spontaneous combustion and the heat liberated maintains the oil at the desired temperature.

A pressure of 150 to 180 pounds is maintained on the system and the products from the second drum are cooled by the incoming raw oil. R. P.

454. Apparatus for Treating Oil. C. P. Dubbs. U.S.P. 1,670,104, 1928.

This invention relates to improvements in apparatus for treating oil and refers more particularly to an apparatus for pressure distillation in which the oil is treated in a battery of stills, any number of which stills may be in operation or on stream at any given time.

From five to twenty stills or more may constitute a battery. These stills are mounted in suitable furnaces whereby they may be independently heated. Each still is provided with a residue draw-off pipe having a throttle valve and a pressure gauge. Oil is introduced into the stills by means of a pump, the inlet side of which is connected to raw oil supply. The discharge side of the pump is connected to a delivery header. This delivery header is provided with branches extending into each still near the lower end of the latter, this extension being perforated in order to discharge the fresh oil along the entire length of the still. The arrangement is such that, by suitably regulating the valves, fresh oil may be fed to one or more of the stills. Each still is provided with a vapour outlet pipe connecting to the lower end of a header. This header is provided with a reflux condensate draw-off pipe connected to the suction side of the pipe. Each vapour pipe is provided with a throttle valve by which the pipe may be entirely closed, and with automatic reducing valve and check valve, the latter tending only to permit the vapours to pass into the lower header. This header is connected to a plurality of upwardly inclined aerial condenser pipes, the upper ends of these pipes being in turn connected to the bottom of a second header. Throttle valves are interposed in either end of each pipe whereby any given pipe may be cut out of the system in case there is an excess of aerial condensing surface. The upper end of the second header is connected to the top of a condenser coil, which is connected to a receiver. This receiver is provided with pressure gauge, liquid level gauge, distillate draw-off and gas outlet pipe.

The process may be operated by charging, say, three stills from one-half to two-thirds full of gas oil, and heating until an oil temperature of 700° to 750° F has been reached and the stills are under a vapour pressure of 135 pounds. The valves are then opened and the vapours permitted to pass through the condensing part of the apparatus. The pressure will then be regulated by means of the valves on the receiver. The automatic reducing valves are so set as to maintain a difference in pressure of, say, 10 pounds;

while the pressure on the still is 135 pounds, the pressure on the condensing part of the apparatus is 125 pounds. Inasmuch as only part of the battery is now in operation, the remainder of the stills are cut out of the system by closing the proper valves. There will now be an excess of aerial surface, so a number of pipes are cut out of the reflux condenser by closing their corresponding valves. As the vapours pass through the inclined aerial lines, the heavier fractions will condense and be returned to the bottom header by gravity and thence delivered to the feed pump. Fresh oil may be continuously fed in regulated quantities to the stills of the battery which are in use, and the residue constantly drawn off. In this way, the process may be continuous. If desired, the charging stock may be fed intermittently or the process may be operated as a batch process. R. P.

455. Apparatus for Treating Hydrocarbons. G. Egloff. U.S.P. 1,670,105, 1928.

This invention relates to an improved apparatus which gives a maximum heating area in relationship to the amount of oil treated at any given time, in which the oil vapours can be superheated, and if desired again subjected to additional heating.

Five horizontal cylindrical stills are arranged so that two are superimposed above two and the fifth is superimposed medially above the others. The lower pair of stills are connected near the bottom to each other and each is connected at the top by three straight ducts to the bottom of the still immediately above. The upper pair of stills are again connected to the fifth still by ducts which connect the topmost still below the middle line to the top of the stills beneath.

The vapours are taken from the top still through a dephlegmator to a condenser. The raw feed is also pumped into this still.

In operating the plant, the oil is charged into the stills until all are full excepting the top still, which is filled to about half its capacity when operating on waxy oils which are more easily cracked. With oils which are more difficult to crack, only the bottom two stills are charged fully. In this case, the upper stills act as superheaters of the vapour. R. P.

456. Apparatus for Cracking Oil. G. Egloff. U.S.P. 1,670,106, 1928.

This invention relates to improvements in apparatus for cracking oil and refers more particularly to an apparatus for conserving heat and securing the maximum amount of pressure distillate from the original charge. In the art of cracking oil, the generated vapours contain a large percentage of incompletely cracked condensates which have to be subjected to reflux condensing action and subjected to further cracking. Under present practice, there is a very large heat loss due to the necessity of frequently condensing these heavier vapours and again reheating them. In the present invention all of the reflux condensation takes place in the body of the still itself, this reflux condensation being produced by the cool incoming oil, which oil is also preheated by the generated vapours before it enters the main body of oil in the still.

The apparatus is composed of a still situated over a furnace. Within the still an open pipe is concentrically supported in a slightly larger pipe forming a jacket tube. This tube runs horizontally in the upper part of the still and extends practically to its full length. From one end of the jacket a pipe extends downwards and connects to a perforated tube running along the bottom of the still. The other end of the jacket is connected to the feed pump. The raw oil is continuously fed through the narrow chamber formed by the

jacket of the open-ended pipe, thence through the perforated tube into the body of the still. Thus the vapours rising from the oil are cooled by the incoming raw oil, the heavier fractions are condensed and fall back into the liquid, whilst the cold oil is preheated at the same time. R. P.

457. Process of Cracking Petroleum Oil. G. Egloff. U.S.P. 1,670,108, 1928.

In a plant of the Dubbs type, oil is heated in a coil and expanded into a large chamber. The residium is drawn from the chamber and the vapours pass into a large horizontal dephlegmator. The residue from the chamber and the reflux from the dephlegmator may be re-circulated through the expansion chamber by a pump, or part of each may be re-circulated and part may be by-passed out of the system. The uncondensed vapours from the dephlegmator are cooled and collected in a receiver. R. P.

458. Distillation of Oils with Catalysts. G. L. Pritchard and H. Henderson. U.S.P. 1,672,339, 1928. Appl., 28-11-1921.

High-boiling oils are distilled in presence of aluminium chloride until not less than one-tenth gallon and not more than 5 gallons of gasoline are obtained per pound of aluminium chloride used. The latter, admixed with oil, is then withdrawn and distilled, the aluminium chloride so separated being returned to the cracking still. H. G. S.

459. Pressure-Still Process. C. B. Buerger, assignr. to Gulf Refin. Co. U.S.P. 1,672,801, 1928. Appl., 26-4-1927.

The residue draw-off pipe from the reactor used in cracking oils under pressure tends to choke owing to deposition of tarry matter and coke. This is prevented by periodic flushing with a solvent oil—*e.g.*, the pressure distillate from the process. H. G. S.

460. Process of Cracking Mineral Crude Oils. H. Blumenberg, junr. U.S.P. 1,673,491, 1928.

The process consists in adding aluminium chloro sulphate to the extent of 2 to 5 per cent. by weight to crude mineral oil, heating the oil to cracking temperature, distilling the oil and condensing the distillate. H. G. S.

Oil Field Technology.

461. Prospecting with Rotary Equipment. C. P. Parsons. *Oil and Gas J.*, 1928, **27** (3), 99, 156.

The factors affecting prospecting with rotary equipment are basic and supplemental, mechanical and human.

The physical reaction of a rotary bit, and its transmission to the surface *via* the drill-pipe, are the first of a series of combined indications as to the nature of the formation. Speed of cutting is also an indicator, as is also speed of circulation. The cuttings are also another source of identification, and the markings on the cutting edge of a fishtail bit serve as a check.

Where absolute assurance is necessary, doubt can be removed by coring, and it is in the use of core barrels that rotary has progressed in prospecting. There are two types of core barrel, single and double, the latter being somewhat expensive and complicated.

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Where an oil or gas sand is to be tested without the expense of running a string of casing, the hole is reduced at about the top of the sand, and the sand penetrated with a double-tube corer.

Present-day testers have a conical packer which sets on the shoulder and shuts-off the mud outside the stem, which is run in closed and empty. When seated, the tester is opened and any gas will blow through the drill stem and show on the derrick floor. Before raising, the tester is closed to retain any oil or salt water from the sand.

A hole which stands up well is necessary to successful coring and where local mud is not sufficiently heavy to ensure this, minerals such as barites, hematite, etc., are added.

Differential controls are simple in operation, and provide for the proper weight being on the bit at all times.

In rotary prospecting the human factor is of vital importance. G. S.

462. Engineer on Production Economics. S. F. Shaw. *Oil and Gas J.*, July, 1928, **27**, (3), 38, 105.

In the early days of a large well, gas-lift costs are merely nominal, ranging from 1 to 5 cents. per barrel. Later, when more input gas is necessary, costs increase in proportion. For pressures over 100 lb./sq. in. a two-stage compressor is required, costing about 4 cents. per 1,000 cu. ft. when using fuel gas from the lease and 7½ cents. per 1,000 cu. ft. when using an electric motor with current at 1 to 1½ cents. per kw. hour. For pressures under 100lb. a single-stage compressor with free fuel gas will cost about 2½ cents. per 1,000 cu. ft. and 5 cents. with an electric motor.

In wells over 4,000 feet deep, the casing diameter usually necessitates input gas pressures over 100lb. With a consumption of 800 cu. ft. of gas per barrel in wells making 1,000 barrels per day, the cost will be 3·2 and 5·6 cents. for gas and electric drive, respectively. At a production of 300 barrels per day, input gas will be about 3000 cu. ft. per barrel, and costs 12 to 21 cents., respectively. In deep wells with small productions, the input gas may reach 10000 to 15000 cu. ft. per barrel. Even then the cost of 25 to 37½ cents. per barrel may still be lower than by beam pump or pumping jack.

In some cases, the cost is offset by the value of gasoline produced during compression. Between 300 to 500 barrels, tapered tubing can be installed and input gas reduced to about 2000 cu. ft per barrel. At about 100 barrels, single-stage compression could be used with a consumption of about 5000 cu. ft. per barrel. Where oil and water are produced, the consumption of gas per barrel is greater than for oil alone.

In Oklahoma, gas lift has been used in 65- to 500-barrel wells at 2800 feet input pressures ranging between 32 and 50lb.

Where gas lift is employed, periodical tests should be made to determine the proper rate of production. A regular schedule of inspection and repairs to apparatus should be followed.

Flow nipples have been used in many fields to reduce gas-oil ratio, but restriction of flow appears to hold back a larger proportion of oil than of gas. G. S.

463. Engineering in Natural Gas Industry. E. L. Rawlins. *Oil and Gas J.*, 1928, **27** (3), 111, 144, 147, 148.

Much advance has been made in methods of removing water from gas wells other than by blowing, particularly with siphons, bailing machines and swabbing lines, and it is recommended that casing in a gas well should always be cemented.

Conservation of natural gas is being helped by better knowledge regarding open-flow gauging and it is recommended that all open-flow tests be based upon a standard opening.

A system of back pressure control has been established which permits lower line pressures for operation, generally less than 56 per cent. of the working pressure on the well.

Specific gravity is important in gas measurement, particularly with orifice meters, where the quantity of gas varies inversely with the square root of the gravity. Much work has been directed towards securing accurate gravity determinations.

The question of transmission losses is also receiving attention from the American Gas Association, and the Bureau of Mines, who are also studying the flow of gas through pipe-lines. G. S.

464. Natural Flow and Gas Lift Experiments. J. L. Dwyer. *Oil and Gas J.*, 1928, **27** (3), 216–217.

The equipment of the Bartlesville Station of the U.S. Bureau of Mines for flow and gas lift experiments was described in a previous article (see Abstract No. 365), and the present article gives a more technical discussion of the work.

Success will depend on the accuracy of measurements of the oil and gas involved, and in some cases triple checks will be made.

Natural gas from the city mains will be measured both by an orifice and a displacement meter, and compressor input gas will be measured by displacement meters. The gas will pass through a temperature control system from the compressors to the high-pressure receivers, the discharge from which will be measured by displacement meters. An after cooler will be installed ahead of the high-pressure receivers. Temperature changes in the reservoir will be negligible owing to its size.

In the reservoir, pressures will be taken in the sand near the well and in the well itself, and the differential pressures recorded carefully.

Tubing will be of high-grade steel, $\frac{1}{4}$ to 2 inches in diameter, and the flow lines will be fitted with transparent sections for observing the flow.

Oil from the separator will be accurately gauged and the gas will pass through a drip before being measured in a displacement meter.

Solubility tests of the gas in the oil will be made at varying pressures and temperatures, and the selective solubility of the various constituents will be determined. Compressibility tests on the gas will determine the deviation from Boyle's law.

Before pumping the gas-saturated oil into the reservoir, the latter will be charged with gas at 200lb., and this pressure will be maintained on the reservoir by a back-pressure valve during charging.

When fully charged, the reservoir will contain approximately 75 bbls. of oil, and about 46 cu. yards of sand. The starting input of oil will be at 40 gallons per minute, and it is estimated that possibly 8 hours will be required fully to charge the reservoir.

Illustrations of the apparatus are given. G. S.

465. Cable Drilling and Fishing Tool Code. *Oil and Gas J.*, 1928, **27** (3), 118, 159, 160, 163.

The A.P.I. Committee has adopted 11 sizes of cable tool joints, 7 threads to the inch, as follows: $1\frac{1}{2}\times2\frac{1}{2}$in., 2×3in., $2\frac{1}{2}\times3\frac{1}{2}$in., $2\frac{1}{2}\times3\frac{1}{2}$in., $2\frac{1}{2}\times3\frac{1}{2}$in., 3×4in., $3\frac{1}{2}\times4\frac{1}{2}$in., $3\frac{1}{2}\times4\frac{1}{2}$in., 4in.$\times5$, $4\times5\frac{1}{2}$in., $4\frac{1}{2}\times6$in. During transport all tool joints should be protected and all working parts shielded,

Before making up a string, all joints should be carefully examined and faulty or damaged ones rejected. After lubrication, the joints are made with slight pressure on the jack. The joints are then broken, examined, lubricated and again set up with more pressure. They are again broken, cleaned, lubricating oil removed and again made with still greater pressure. After again breaking and cleaning the joint is then set up ready for use.

The dressing and hardening of drilling bits require careful study. Before placing in the furnace any frost should be removed and bits should always be subjected to slow heating to ensure uniform expansion. Care should also be taken that the correct fuel-air mixture is used. For the dressing of bits it is suggested that mechanical means offer considerable advantages.

After dressing, the bit should be allowed to cool to about 900° F. (black) before hardening. On reheating, the critical temperature is 1450—1500° F. (medium to dull cherry red), and when this is reached, the bit should be quenched in water at between 50—100° F. The use of a slack tub with a circulating system is recommended.

Under-reamer cutters are treated in the same way as bits, but are normalised prior to hardening. After cooling until black they are heated in a rich fuel mixture to about 1550° F. (medium cherry red) and cooled by slow quenching, preferably in oil. Hardening is then proceeded with as usual. This treatment applies to carbon steel; for special alloys, the temperatures will have to be varied accordingly.

The oxy-acetylene method is often used to build up under-reamer cutters with special alloys, and if properly applied lengthens the life of the cutters.

The diameter and length of all tools should be carefully logged for reference in the event of a fishing job.

In fishing, it is important to ascertain the position and size of the fish. The running of an impression block and reference to the log should determine this. Fishing tools should be of accurate size, and of the best grade and design.

All tools should be periodically inspected and oiled and all defects plainly marked. G. S.

466. Hammer Drill not Suitable for Wells. L. G. E. Bignell. *Oil and Gas J.*, 1928, **27** (3), 100, 244–246.

The energy developed by the falling string of tools in cable drilling is in effect a form of diametrical crushing, while the action of a cone or roller bit in rotary drilling is radial crushing. The rotary fishtail bit exerts a form of shearing force.

Considering the hammer system of drilling, as used in the mining industry, it would seem that this type of equipment cannot be applied to drilling deep vertical holes. The nearest attempt to apply hammer drill principles is in the Fauck system, but this does not compare favourably with the combination outfit using rotary and percussion. G. S.

467. Pipe Stresses Caused by Temperature. *Oil and Gas J.*, 1928, **27** (3), 103, 143.

Where possible it is important that pipe-lines should be buried to a sufficient depth to minimise fluctuations in temperature.

When subjected to temperature change, pipe will vary in length, the formula for this change being

$$l_1 = l(1 + \phi t)$$

when l_1 = length at final temperature,
l = length at initial temperature,
ϕ = coefficient of expansion,
t = degrees of change.

The value of ϕ is 0·000012 per °C., and 0·0000067 per °F. for wrought iron, 0·0000065 per °F. for steel, and 0·0000062 per °F. for cast iron, the modulus of electricity for wrought iron being 27,000,000, for steel 30,000,000, and 15,000,000 for cast iron.

It is necessary to allow sufficient "slack" in a line for changes in length due to temperature changes, and a chart is given showing the necessary offsets from the centre line of the pipe ditch to allow a definite "slack." A chart is also given, showing the relation between deformation in inches per 100 feet and temperature changes in °F. The curves for this chart are based on the above formula or

$$l_1 - l = l\phi t.$$

A good safe practice is to allow twice the calculated "slack."

A chart is also given showing ultimate strengths and working stresses for steel, wrought iron and cast-iron pipe. G. S.

468. Proper Application of Pipe Coatings. *Oil and Gas J.*, 1928, **27** (3), 39, 96, 177, 178, 181.

Before laying a pipe-line, the corrosive characteristics of the soil and soil-waters should be studied. As a rule primary alkaline waters are non-corrosive, while secondary alkaline, secondary saline, or even acid waters are corrosive.

Bituminous pipe coatings are generally made from native or natural asphalts, residual asphalts, coal tar pitches and other natural hydrocarbons.

About 90 per cent. of the failures of bituminous pipe coatings are caused by improper application of the coating.

The vital factors determining success or failure of pipe coatings are: (1) Quality in relation to impermeability and durability, (2) Proper application to the pipe, (3) Adhesion of coating to pipe, (4) Handling of pipe after coating to avoid damage.

The primary purpose of pipe coating is to exclude water from contact with the pipe, and a good coating should last from 10 to 15 years. The primary requirements for success are: (1) Use of good material and strict adherence to instructions for use, (2) The exclusion of foreign matter from the coating material, (3) A clean, dry metallic surface upon which to apply the coat, (4) a firm, tight band between coat and pipe, (5) A uniform coating of the pipe and freedom from air bubbles, etc., (6) Adequate thickness of coating to afford the necessary protection. G. S.

469. Recent Improvements in Fishing Tools and Devices used in Rotary Drilling. Bernard H. Scott. *The Oil Age*, Vol. 25, No. 2, page 23.

This article gives a short description of the construction and use of several of the tools used in modern fishing jobs. The following tools are described:—(*a*) Trip Overshot, an overshot tool which may readily be detached from the fish if the latter proves immovable. (*b*) Packing Off Overshot, an overshot tool containing a rubber gland capable of making a water-tight joint on the broken end so that circulation may be established to loosen a tightly lodged fish. (*c*) Offset Tool Connection, a short hinged connection to be used for connecting the fishing tool proper on to the drill pipe; the hinge action is controlled hydraulically from the surface. (*d*) Hydraulic Wall Hook, a device for reaching out to the side of the hole and securing a broken tool. This device can also be released if required. (*e*) Spiral Guide, another device for catching up drill pipe from caves. The guide is of "corkscrew" pattern and is fixed over an overshot or tap, which latter actually does the gripping. (*f*) Mechanically Operated Grab, a new design of grabbing tool, one feature of which being that the circulation through the drill pipe is shut off whenever

the jaws completely close, thus acting as a tell-tale as to whether the grab has engaged the fish or is empty. (*g*) Rotary Jars, devices for applying a severe jarring action to a firmly embedded tool. (*h*) Safety Joint, a short connection to be employed immediately above the fishing tool and intended for use only in case the broken piece remains immovable. The joint contains a coarse square thread coupling designed not to jam ; this joint may readily be broken if it is desired to disconnect with the fish and save the complete drill pipe. E. R. B. S.

470. Dewatering of Gas Wells. M. Walker. *Oil and Gas J.*, 1928, **26**, No. 32, page 257.

The method of dewatering gas wells by the use of a "siphon" is set out and explained in detail, calculations and formulæ for application to various cases being included. The siphon used consists of a string of tubing of about ¾ inch or 1 inch diameter run into the well to below water-level. The tube has water inlets at the bottom and one or more jet holes situated somewhat above the water surface in the well for the inlet of the gas. The action is that of the ordinary gas lift, and with suitable conditions proves very economical to run. Three examples of the use of such a siphon are quoted, and the wells concerned deliver from 3·5 to 33·3 gallons of water per hour. In most cases the gas used to lift the water may be collected and delivered to the main receiving line. E. R. B. S.

471. Late Developments in Electric Method of Locating Depth of Water Intrusion in Oil Wells. J. H. Tinkham. *The Oil Age*, 1928, Vol. 25, No. 3, page 27.

A short account of the method developed and offered as a field service by the Loomis Oil Well Control Co. for locating accurately the point at which water is entering a well. Measurements are taken of the electrical potential between two dissimilar electrodes lowered into the well on a twin conductor cable, a measuring device being incorporated with the winch so that the exact depth of the electrode may always be known. A series of measurements are taken as follows, the readings being recorded, for preference, in the form of a curve :—(1) Potentials are taken at a series of points throughout the depth of water (at normal level) in the well. (2) An electrolytic solution is distributed evenly through the well, still at normal level, and potentials measured to check this even distribution. A special form of bailer is used for spreading the electrolyte. (3) Some water is bailed from the well so that more water will flow in from the formation, and so dilute the electrolyte. Potential readings taken down the well will now show a marked fall at the point where water has entered. This group of readings is repeated for checking purposes. (4) As a further check fresh water may be run in from the top of the hole, allowing the electrolyte to be displaced through the water entrance. The surface of contact between fresh water run in and electrolyte is followed by the electrodes, and falls at a steady rate until the water entrance is reached, below which there is little or no fall. If water is encroaching at two or more points there will be a sharp reduction in the rate of fall of the dividing surface as each leakage point is passed. The method is rapid and entirely without detriment to the well or casing, the electrolyte used being selected for each case with a view to giving no risk of injury to the well or its surrounding sands. E. R. B. S.

472. Testing High Pressure Gas Meters. John C. Diehl. *Oil & Gas J.*, 1928, **27** (3), 139–140.

Many problems arise in the testing of large-volume high-pressure meters

and the precautions to be taken. The methods which may be used are: (1) Bell prover in shop at low pressure; (2) Test Meter in field at low pressure; (3) Funnel prover or flow meter in field at low or high pressure; (4) Critical flow orifice prover at high pressure.

When meters are removed from the place of use to the shop for testing, they should be allowed to remain for several hours to attain shop temperature. Air should be passed through the meter at the maximum rate for at least 10 revolutions and the meter should then be tested at a rate less than 10 per cent. of the working capacity and at maximum capacity. Where the prover is too small, an extra large hose should be used so that the pressure drop between prover and meter is not over 2 inches of water.

Where meters are too large to be easily moved, a smaller proved meter may be used for comparison purposes *in situ* at low pressures. If correct at three or four rates it is assumed to be correct at the higher pressures.

The funnel or flow meter is well known. With this it is necessary to know the barometric pressure, and specific gravity and temperature of the gas.

Critical flow meters may be used when the pressure is 15 lbs. or more and where the gas can be discharged into the atmosphere or a low-pressure line.

Tables are given of results of passing high-pressure air through orifices, and it is noted that the time for 1 cu. ft. is the same, regardless of pressure ($\frac{1}{4}$ in. orifice, 18 secs.; $\frac{1}{2}$ in. orifice, 4.5 secs.), at the same temperature.

The rate of flow through an orifice in cu. ft. at line pressure, round edge upstream, is constant, when the absolute upstream pressure is twice or more than the absolute downstream pressure.

The formula for obtaining the correct passage time for 1,000 cu. ft. gas through a meter and critical orifice is:

$$t_g = Mt_a \sqrt{\frac{520\ YG}{T}}$$

where t_g = correct time in secs. at line pressure and temperature.
M = index reading.
t_a = time in sec. for 1 cu. ft. air at 60° F.
520 = absolute temperature corresponding to 60° F.
Y = ratio of specific heat of air divided by ratio of specific heats gas.
G = sp. gr. of gas.
T = absolute temperature of gas in °F.

Tables are given for finding factors not known. The gas temperature should be measured at the inlet to the orifice, and the pressure on the field meter should be maintained constant. The duration of the test should not be less than 100 seconds. G. S.

473. Oilfield Emulsion Treating Means. F. Gardner. U.S.P. 1,668,766, 1928. *Appl.*, 24–4.1925.

A process and apparatus for producing improvements in the treatment of oil field emulsions, in order to separate water from crude oil in a simple and economical manner is here described. The effluent is heated prior to separation whereby its viscosity is reduced; the flow of the effluent is then retarded when demulsification is assisted and separation accentuated. A chemical may be added to soften the water in the oil.

The apparatus for producing these improvements consists of the following essential parts:—

A settling tank to effect a primary separation of oil from water, a water discharge from tank, a filter comprising a lower water collecting chamber, a filter section above this chamber, a spray head in the water chamber, a

conductor leading from upper part of settling tank to the spray head, a discharge pipe leading from the upper part of the filter section, a water draw off leading from the chamber and means for introducing a chemical into the conductor by a gravity feed at the discharge from the settling tank.

H. E. T.

Coal and Shale.

474. A General Review of Low-Temperature Carbonisation. F. S. Sinnatt. *Fuel*, 1928, VII, 305–329.

After discussing developments in the coke-oven and gas industries in the way of providing smokeless fuel—coke and gas—to supply domestic requirements, and the probable effect on the future price of various grades of coal, the author describes briefly those processes of low-temperature carbonisation which are either operating on a large scale in this country or are in course of erection. These include externally heated retorts in which the mass of coal is stationary during carbonisation—*e.g.*, the Parker, Illingworth, Hird and Plassman retorts, in which the coal is moved intermittently, *e.g.*, Fuel Research Plant, or in which it moves continuously ; *e.g.*, the Crozier, Fusion Rotary, Freeman Multiple and Salermo retorts. The K.S.G. and Dvorkovitz processes employ combined internal and external heating. Carbonisation by internal heating is adopted in the "L & N" horizontal rotating retort and in the Maclaurin, Midland Coal Products, Sutcliffe and Turner processes, the last four of which use vertical retorts. The results of tests carried out on five plants by H.M. Fuel Research staff are then described in detail as well as data submitted by firms operating six other types of retort. Analyses of the raw coals used and of the semi-cokes produced are tabulated. The yields and properties of the liquid products are next dealt with. The yields of dry tar vary from 16·4 gals. to 50·2 gals. per ton of coal, the latter however being the result of carbonising a cannel coal. The average yield from bituminous coal is about 19 gals. per ton. It is not possible to ascertain the relationship of the composition of the tars to the method of carbonisation employed. The broad groups of constituents can be distinguished fairly easily, but the individual constituents cannot be determined accurately. The effect of increased temperature of carbonisation is to reduce the content of saturated hydrocarbons in the distilled oil. Further, tar made below 600° C. contains only small proportions of benzene and its homologues, no naphthalene but methyl-naphthalenes and methyl-anthracenes. Tar made above 600° C. contains both toluene and naphthalene but only traces of benzene. The hydrocarbons are composed of paraffins, olefines, naphthenes, unsaturated polymethylenes, etc., but no reliable methods of analysis have yet been devised. The phenolic compounds are made up of phenols and its homologues and rhetinols (*cf.* Edwards, *J.S.C.I.*, 1924, **43**, 143). The latter form from 9 to 15 per cent. of the tar from externally heated processes and are complex in constitution. Basic compounds vary from 2 to 4 per cent. of the tar. They are dark coloured, viscous oils, soluble in organic solvents, and are stated to have deleterious effects upon any oils which might be used as high-class lubricants.

One of the valuable products recoverable from primary tar is motor-spirit, which can now be satisfactorily refined and is an anti-pinking spirit. Lubricating oils have been produced from a bastard cannel coal by the "L & N" process, and indications of the presence of lubricants were noticed in a tar from the Leigh Smokeless Fuels Process. Paraffin waxes have also been

isolated by a solvent method due to G. T. Morgan. The residual pitch differs from ordinary pitch, and new uses must be sought for it. Externally heated retorts give a gas of high calorific value, whereas large volumes of weak gas are obtained from internally heated retorts. The former may be used to enrich ordinary town gas or the unsaturated hydrocarbons contained in it may be employed for the manufacture of glycol, etc. The utilisation of the aqueous distillate is also dealt with. The paper concludes with descriptions of a number of processes compiled by the operators or inventors. H. G. S.

475. Some Facts Regarding the Low-Temperature Carbonisation of Coal. G. S. Haslam and R. V. Wheeler. *Fuel*, 1928, VII, 292–4.

The main object of carbonising coal at low temperatures was initially the provision of a smokeless household fuel and more recently the production of home supplies of fuel oil. The authors think that these aims may be ultimately realised, but that the most efficient utilisation of colliery slack is the immediate objective. To produce household fuel, coking coals containing not more than 8·5 per cent. of ash can be employed, necessitating cleaning of the slack. Non-caking coals, unless blended, give soft friable chars suitable for boiler firing on chain-grate stokers or as pulverised fuel, but not for household purposes. Consequently, since ash content of the coke is not of great importance, non-caking slacks need not be washed prior to carbonisation. An average British bituminous coal will yield 15 to 18 gals. of crude primary tar per ton. The tars contain the same essential constituents—*i.e.*, saturated and unsaturated hydrocarbons, phenolic compounds and bases, but not necessarily in the same proportions, whilst the character of the tar from a given coal will vary widely according to the system of carbonisation adopted. Chief interest centres in the hydrocarbon fraction, which contains small proportions of motor-spirit, together with kerosine, fuel oils and occasionally paraffin wax. If the tar is uncracked, it may contain small amounts of high-quality lubricating oils. Cracking may be adopted to enhance the value of primary tars when motor-spirit has a high market value. The average yields of gas at 450° C. and 600° C. are 3000 and 4000 cu. ft. respectively, the respective gases having calorific values of 950 and 700 B.Th.U. per cubic foot. The technical problems met with in carbonisation are concerned with the low heat-conductivity of coal and the tendency of some coals to swell. The former is overcome in some processes by utilising internal heating by means of hot gases, and the latter by blending or by partial pre-oxidation below the temperature at which the coal becomes plastic. H. G. S.

476. Improvements in the Production of Low Boiling Point and other Hydrocarbons by the Destructive Hydrogenation of Coals, Tars, Mineral Oils and the Like. I. G. Farbenind. A.-G. E.P. 273,337, 1928.

In the destructive hydrogenation of tars, mineral oils, etc., it is difficult to select the conditions of temperature, pressure and time of reaction for the production of the desired materials owing to the generally complicated nature of the raw stock. This difficulty can be obviated by treatment at high pressures and temperatures in presence of hydrogen, of fractions of the raw materials having comparatively narrow boiling-point ranges. These particular fractions, preferably of the type of middle oils boiling between 250° and 300° C., may be prepared by distillation, cracking or hydrogenation of liquid raw materials or by dry distillation, low-temperature carbonisation, hydrogenation, etc., of coal and the like. The process is carried out with the aid of catalysts, those of a porous character like charcoal, silicates, hydrosilicates or active silica being advantageous. H. G. S.

477. Improvements in the Manufacture and Production of Valuable Liquid Hydrocarbons and their Derivatives, particularly those of Low Boiling Point, from Coal, Tars, Mineral Oils and the like. I. G. Farbenind. A.-G. E.P. 272,556, 1928.

The liquefaction of bituminous materials, tars, oils, etc., by hydrogenation under high pressure is carried out with flowing gases, preferably in closed circulation, and maintaining a large excess of hydrogen, so that the partial pressure of the resulting products does not exceed 10 per cent. of the total pressure in the reaction chamber. The reaction may be carried out in presence of catalysts and the temperature regulated by means of the excess of gas containing hydrogen. In Example I, crude oil is atomised or gasified in a stream of heated hydrogen in a vertical high-pressure reaction chamber lined with aluminium and is passed at 475° C. and 200 atmos. pressure over a catalyst composed of molybdenum and chromium oxides. The proportions of materials are regulated so that for each cubic metre of cooled product 15,000 cubic feet of gas are maintained in circulation by the pump. The product contains about 60 per cent. of benzine, the formation of methane being practically nil. Example II quotes the production of 80 to 85 per cent. of liquid from coal, the latter being made into a paste with heavy anthracene oil and 1 per cent. of bauxite, and treated so that for each litre of coal paste there are circulated 12 to 15 cubic metres of hydrogen. The reaction chamber is vertical and its hot parts are lined with aluminium.

H. G. S.

478. Improvements relating to the Distillation of Coal and other Solid Carbonaceous Materials. T. A. Goskar. E.P. 291,836, 1928.

Finely ground bituminous coking coal is intimately mixed with an agglomerating agent consisting of a solid hydrocarbon of the paraffin or terpene series—*e.g.*, bitumen or resin, the resulting mass being then intermingled with finely ground coal waste, anthracite duff, coke breeze and the like and distilled. The charge, before distillation, may be heated to about 125° to 150° C. under a pressure up to 40 lb, then cooled to about 70° C. and briquetted. The proportions of the various materials are specified as 20 to 40 parts by weight of caking coal, 8 to 15 parts of bitumen or resin, and 70 to 50 parts of carbonaceous material. Distillation of the mixture, whether briquetted or not, is preferably effected in a vertical or inclined retort internally heated by gases which do not support combustion of carbon—*e.g.*, producer gas, water gas or steam, air being rigorously excluded. H. G. S.

479. Method of Carbonising Coal. S. Dunlop for M. J. McQuade. E.P. 291,962, 1928.

Coal is subjected to low-temperature carbonisation in an externally heated retort in such a way that the material is caused alternately to advance a predetermined distance and then to be withdrawn through a less distance. The retort is maintained less than full of coal, the volatiles which collect in the space above the body of coal being withdrawn as they accumulate. The retort, which is cylindrical and horizontal, is fitted with a co-axial screw conveyor driven by mechanism which causes it to rotate a certain distance in one direction and then to rotate a less distance in the opposite direction. The material is thus continually agitated, is prevented from sticking to the walls of the retort, and all particles are brought into contact with heated surfaces, whilst the sum of the forward and rearward movements enables a shorter retort to be used. H. G. S.

480. Improvements in or relating to the Distillation of Solid Carbonaceous Materials. H. Nielsen and B. Laing. E.P. 292,060, 1928.

Solid carbonaceous materials, preferably of a coking character, are subjected to the action of hot gases containing a controlled percentage of oxygen so as to influence their coking power, and are then subjected to carbonisation. The hot gas used for the treatment may be combustion gas containing from 2 to 8 per cent. of free oxygen. The carbonisation at low temperatures, with by-product recovery, may be followed by carbonisation at a high temperature, the resulting coke being then gasified either with steam, free oxygen, or the combined oxygen present in metallic ores, such as iron or copper oxides. H. G. S.

481. Improvements in the Production of Stable Suspensions or Pastes of Coal. I. G. Farbenind. A.G. E.P. 292,217, 1928.

The process consists in mechanically dispersing finely ground coal in alcohols, using inorganic or organic substances of basic character soluble in the alcohols as stabilising additions, with or without the addition of a minor amount of mineral oils. The alcohols may be methanol or isobutyl alcohol, such as may be obtained by the catalytic reduction of oxides of carbon, while the stabilising media may be the hydroxides of the alkali metals, ammonia, methylamine, pyridine, aniline and the like. Three examples are given. H. G. S.

482. Treatment of Solid Bituminiferous Material. W. H. Hampton U.S.P. 1,668,898, 1928. Appl., Aug. 20, 1919.

A mixture of crushed oil-shale and a heavy oil is preheated to from 300°–400° F. to soften the bitumen in the former, ground to a fine state of division, and then digested at a temperature not exceeding 700° F., cracking being substantially avoided. A certain amount of distillation occurs during digestion, which may be facilitated by the introduction of steam or an inert gas or by operating under reduced pressure. The residue is thinned down, whilst hot, with kerosine and then centrifuged, the residue being thoroughly washed with kerosine and dried. Higher yields of oil than are obtained by dry distillation of the shale are claimed. H. E. T.

483. Process of Treating Shale or the like. H. D. Ryan. U.S.P. 1,672,231, 1928.

Bituminous shale or the like is simultaneously ground and digested with a hydrocarbon oil at temperatures sufficiently high to liquefy shale bitumens, limiting vaporisation so as to obtain a final digestion product which is fluid. The residual solid matter is then removed. The temperature at which the shale is ground in contact with the oil, preferably a heavy hydrocarbon oil, ranges from 300° to 400° F., but in certain cases the temperature may be increased to a maximum of 700° F.—*i.e.*, digestion and grinding may take place simultaneously. H. G. S.

484. Process of Separating the Neutral Oils in Tars, Tar Oils and Pitches from Acid Constituents. H. Wittek. U.S.P. 1,674,710, 1928. Appl., 14-4-1927.

The neutral oil and acidic fractions of tar oils, etc., are separated by mixing the vapours of the tar, of an organic solvent and of an oil composed mainly of hydrocarbons of the paraffin series, agitating and then condensing the vapours and separating the resulting layers. The organic solvent in which the acidic portion of the tar dissolves may be ethyl alcohol or any other solvent not miscible with the hydrocarbon oil. H. G. S.

ABSTRACTS.

Arranged by H. G. SHATWELL, Ph.D., and W. H. THOMAS, A.R.S.M.

Abstractors.—S. F. BIRCH, Ph.D., W. S. CLARKE, B.Sc., P. DOCKSEY, B.A., W. H. FORDHAM, M.A., F.G.S., H. S. GARLICK, B.Sc., W. N. HOYTE, B.A., W. S. NORRIS, Ph.D., R. PITKETHLY, A.I.C., G. SELL, H. G. SHATWELL, Ph.D., R. STANSFIELD, A.M.Inst.C.E., A. R. STARK, B.Sc., H. E. TESTER, B.Sc., T. WISKIN.

General.

485. IMPROVEMENTS IN THE MANUFACTURE OF MINERAL OILS FROM BITUMINOUS MATERIALS. C. W. M. Bervoets. E.P. 270,349. May 2, 1928.

A biological process is described for the manufacture of mineral oils from peat, lignite, coal and other bituminous materials.

An apparatus, consisting of a large covered container, steam heated, with arrangements for forcing ventilating air through the contents and for discharging the products formed, is 90% filled with the raw material mixed with 30–50% of its weight of salt water. The mass is heated to 50° C. and fermented without additional heat by micro-organisms normally contained in the raw materials. During the process the temperature must not be allowed to rise above 80° C. H. S. G.

486. TREATMENT OF SUBSTANCES WITH HEAT. W. G. Laird. U.S.P. Re. 16,971. May 22, 1928. Original Patent 1,546,345. July 14, 1925.

The process claimed consists of mixing a liquid with a gas or a fluid, passing the mixture to a zone of maximum temperature from which vapours are withdrawn. The vapours are passed counter-current and in heat exchange relations with the mixture through a zone or zones in which the mixture is at a lower temperature than in the first zone, the condensates formed being intermingled with the mixture in the second zone. Apparatus for heating and distilling liquids by the process is claimed. G. S.

487. IMPROVEMENTS IN AND RELATING TO APPARATUS FOR THE PRODUCTION OF DISPERSIONS OF SOLIDS IN LIQUIDS. W. H. Whatmough. E.P. 293,510. July 12, 1928.

An improved process and apparatus is described for the dispersion of amorphous aggregates in liquids. The apparatus consists essentially of a liquid tight casing, within which is a rotor provided with one or more beater elements of reticulated structure which can be rotated at high speed. Baffles, which may be of similar structure to the beaters, are employed in order to direct the liquid into the path of the beaters.

By composing the beaters wholly or in part of wire gauze, the amorphous aggregate is subjected to a rubbing action which effectively disperses it throughout the liquid. If the rubbing action is confined to the surface of the gauze, a maximum efficiency is not obtained except by repeated treatment; an effective result may, however, be obtained by allowing the solid

while suspended in the liquid to impinge at high speed on the beaters, the latter being provided with interstices through which the liquid can pass in rubbing contact.

Various forms of the apparatus used in carrying out the process are given in the diagrams, and it is claimed that with this apparatus highly concentrated mixtures such as 3 pints of oil, 1 lb. of wax and 4 lb. of mineral blue can be successfully dispersed by subjecting them to a high speed treatment for ten minutes at an initial temperature of 100° C. H. E. T.

See also Abstracts Nos. 511, 512 and 599.

Chemistry.

488. Thermal Decomposition of Ethane, Ethylene, Propane and Propylene. F. E. Frey and David F. Smith. *Ind. Eng. Chem.*, 1928, **20**, 948–951.

An account is given of some experiments on the thermal decomposition of ethane, ethylene, propane and propylene. It was found that propane is decomposed very rapidly in the presence of a nickel catalyst at 350° and 400° C., giving carbon, a large amount of methane and some hydrogen. The quantity of hydrogen formed increased, and the methane produced decreased with increase in temperature. At 200° C. a small amount of methane only is formed. The mechanism suggested for the reaction is:—

$$C_3H_8 \rightarrow 2CH_4 + C$$
$$C_3H_8 \rightarrow C_3H_6 + H_2 \rightarrow CH_2 + 2H_2 + C$$

It is suggested that although the first reaction occurs at all three temperatures, the second occurs at higher temperatures only.

A copper catalyst only slightly effects the decomposition of propane at 350° and 400°, while platinum produces a small but definite effect at 568° C., carbon, as well as hydrogen, methane, ethylene, ethane and propylene being formed.

The only products formed from ethane in silica vessels at 575° C. are ethylene and hydrogen. Other products are only formed when the concentration of ethylene becomes sufficiently great. Under similar conditions the products from propane can be explained by assuming the following reactions:—

$$C_3H_8 \rightarrow C_2H_4 + CH_4$$
$$C_3H_8 \rightarrow C_3H_6 + H_2$$
$$2C_3H_8 \rightarrow C_3H_8 + C_2H_6 + CH_4.$$

Ethylene is much more rapidly hydrogenated than dehydrogenated in silica vessels at 575° C. The speed of hydrogenation is more dependent on the hydrogen concentration than upon the ethylene. Under similar conditions propylene reacts more slowly, but the reaction like that of ethylene is considerably complicated by the formation of polymerisation products.

The polymerisation of ethylene at 575° C. in silica vessels results in a relatively large amount of propylene as well as in the formation of higher hydrocarbons; methane and ethane are also formed. The principal products from the polymerisation of propylene under similar conditions are butylene and higher hydrocarbons as well as methane and ethane. Under the same conditions little if any acetylene is produced from ethylene or its polymerisation products. The presence of hydrogen tends to the formation of more saturated products. S. F. B.

489. Refining Cracked Distillates by Means of Hypochlorite. S. F. Birch and W. S. G. P. Norris. *The Refiner*, 1928, **7**, 94–98.

The action of aqueous hypochlorite solution on sulphur compounds in

petroleum solution, and the influence of excess of alkali, are as follows. Hydrogen sulphide is oxidised to free sulphur, with simultaneous formation of traces of sulphuric acid; this reaction is not affected by free alkali. An organic sulphide is oxidised to the sulphone, which, being neutral, becomes distributed between the petroleum and the aqueous layer. The reaction is qualitatively unchanged by increasing the alkalinity of the reagent, but the oxidation of the higher sulphides is appreciably retarded. A mercaptan is oxidised in two steps. In the first the main product is the disulphide, and the velocity of this first stage in the oxidation of a mercaptan by hypochlorite is practically independent of the alkalinity, although as this is increased there is a tendency to the exclusive formation of the disulphide, Disulphides are not markedly disagreeable in odour and are not reactive to sodium plumbite; hence, although the disulphides remain in the oil, the latter is already "sweetened." The second reaction in the oxidation of a mercaptan is the oxidation of the primarily formed disulphide to the sulphonic acid. This is greatly retarded by the addition of free alkali to the hypochlorite and, in fact, may be almost completely inhibited. Hence desulphurisation is best accomplished by the use of hypochlorite containing only sufficient free alkali to render it stable for purposes of storage, whereas sweetening is possible by the use of solutions containing enough free alkali to prevent the oxidation of disulphides.

This is made the basis of a process of refining cracked distillates by means of hypochlorite. For this purpose the use of highly reactive hypochlorite, containing much free hypochlorous acid, is to be avoided owing to the risk of formation of chlorine derivatives of the reactive hydrocarbon constituents. This risk is minimised by using comparatively strongly alkaline hypochlorite solutions which contain very little free hypochlorous acid and which are non-reactive towards the hydrocarbon constituents, but which are capable, nevertheless, of converting mercaptans to disulphides, thus sweetening the oil. This application of hypochlorite refining to the treatment of cracked distillates is analogous to the use of sodium plumbite and elementary sulphur, but avoids the risk of free sulphur finding its way into the finished product. Further advantages offered by the alkaline hypochlorite process are: desulphurisation brought about by oxidation of sulphides, lower cost than plumbite, absence of sludge, and the fact that no recovery plant is required.

There are two main sequences of treatments in which hypochlorite-, soda- and acid-washing are combined with redistillation: (1) soda-wash, acid-wash, hypochlorite-wash, distillation, soda-wash; (2) soda-wash, hypochlorite-wash, acid-wash, soda-wash, distillation, soda-wash. The second of these gives a product of slightly better odour and is finally adopted. Examples of both methods are given, using a pressure distillate from Persian 60 per cent. residue, cracked distillates produced in a Cross plant from Persian gas oil and from Trinidad gas oil and cracked distillates produced in an autoclave from Roumanian fuel oil, Californian kerosine and Trinidad fuel oil. An initial soda-wash is always given to remove hydrogen sulphide, which otherwise would give elementary sulphur on oxidation. The quantity of alkaline hypochlorite is always regulated, only sufficient being used to sweeten the soda-washed distillate. Chlorination to any appreciable extent is not encountered, and spirits refined by the methods outlined do not corrode copper, do not contain undue qualities of gum or gum-forming constituents, and are satisfactory in colour and odour.

The functions of the refining agents used are found to be quite clearly defined. A finished product of good colour and low gum-forming propensity is only obtainable by the use of an adequate quantity of acid followed by

careful redistillation; the acid used then determines colour, gum formation and sulphur content. The odour of the finished product is affected chiefly by the hypochlorite employed. Increasing the acid treatment gives increased stability towards light. This test, the conditions of which do not in any way resemble the conditions under which the finished commercial product is stored or used, is considered unnecessarily drastic; observation of the behaviour of the material in the dark but in the presence of air would be preferable. W. S. N.

490. Improvements in the Production of Methanol and Other Alcohols and Preparation of Catalysts Therefor. Syn. Amm. and Nitrates, Ltd., and R. G. Franklin. E.P. 293,056. 1928.

Catalysts for the production of methanol from carbon monoxide and hydrogen are prepared by heating a mixture of basic zinc carbonate and basic chromium carbonate. The basic carbonates may be precipitated from a mixed solution of zinc and chromium salts, the solution being prepared by the reduction of sodium bichromate with zinc in the presence of an acid. The catalyst should preferably contain 70 to 80 atoms of zinc to 30 to 20 atoms of chromium. H. G. S.

491. Improvements in the Manufacture and Production of Hydrocarbons and More Especially Liquid Hydrocarbons. I. G. Farbenind Akt.-Ges. E.P. 293,185. 1928.

In the preparation of liquid hydrocarbons by the interaction of carbon monoxide and hydrogen, contact masses are employed which contain elements of the 8th group of the periodic table or their compounds, in addition to copper, silver, gold, zinc in the metallic state or their mixtures or alloys Temperatures of the order of 200° to 400° C. and high pressures—*e.g.*, 200 atm.—are preferably used, and the hot parts of the apparatus should not be composed of iron. H. G. S.

492. Improvements in the Production of Liquid Hydrocarbons. I. G. Farbenind Akt.-Ges. E.P. 269,521. 1928.

Liquid hydrocarbons are produced by passing gaseous mixtures consisting substantially of oxides of carbon and hydrogen over contact masses composed wholly or partially of metallic sulphides, the latter being maintained at an elevated temperature and the pressures being less than 50 atm. The apparatus, especially the heated parts in contact with the gases, must be kept free from iron. Thus, water gas at 20 atm. pressure is passed through an aluminium-lined pressure apparatus charged with cobalt sulphide compressed into lumps, the temperature being 270° C. An oil similar to petroleum, together with water, is obtained on cooling the effluent gases. The catalysts are claimed to be immune to toxic influences. H. G. S.

493. Improvements in the Manufacture and Production of Gaseous Olefines and Liquid Hydrocarbons from Tars, Mineral Oils and Similar Hydrocarbons. I. G. Farbenind Akt.-Ges. E.P. 269,593. 1928.

Gaseous olefines and liquid hydrocarbons are prepared by the interaction of steam and tars, mineral oils, etc., at elevated temperatures, and in the absence of metals which are attacked by superheated steam and which further the formation of carbon dioxide from carbon monoxide and steam. *Example* 1: 3 kilograms of steam and 1 kilogram of refined brown-coal tar are passed per hour at 760° C. through a copper tube 2 metres long and 50 millimetres I.D. The issuing gases are cooled to condense water and

then passed through active charcoal. The products per cubic metre of gas consist of 40 c.c. of a colourless combustible liquid boiling between 30° to 70° C., whilst the gas itself contains 46·2 per cent. of unsaturated hydrocarbons. *Example* 2: Paraffin (B.P. about 360° C.) is vapourized, mixed with 3 to 4 parts of steam and directed through a tube composed of 65 per cent. nickel, 12 per cent. chromium and 23 per cent. iron, and filled with wire spirals of the same material. At 700° C., a gas containing 66 per cent. of olefines is obtained, and this, on cooling, furnished a yellow oil boiling between 80° and 300° C. No deposition of carbon occurs in either of the examples mentioned. H. G. S.

494. Manufacture of Condensation Products from Naphthalene or Naphthalene Derivatives and Ethylene. I. G. Farbenind Akt.-Ges. E.P. 265,601. Feb. 3, 1928.

Condensation products of ethylene with naphthalene or partly hydrogenated naphthalene (*e.g.*, tetraline) are produced by causing ethylene to react under pressure (*e.g.*, 20 atmospheres) with naphthalene or its derivatives in the presence of aluminium chloride or iron chloride at a temperature not below 50° C., preferably in the presence of a halogen acid or of a benzene hydrocarbon. According to the conditions of operation one or more ethyl groups are introduced into the naphthalene molecule; highly viscous, strongly fluorescent oils may be obtained. W. S. N.

495. Process of Treating Petroleums. W. W. Gavy. U.S.P. 1,667,440. July 17, 1928.

A process and apparatus are described, by means of which petroleums, shales and other organic compounds are subjected to controlled oxidisation with nascent oxygen in order to remove impurities such as sulphur compounds, nitrogenous bodies, acids, etc. The nascent oxygen is produced from the anhydride of an oxidising acid, and reacts with the sulphur compounds in the oil producing sulphur dioxide, and at the same time reacting with the other impurities present causing their separation by precipitation.

The treatment of the petroleum is carried out as follows: a theoretical mixture of SO_3 and air is conducted from a gas holder to a drier to remove moisture, and from thence to a catalyst—namely, a platinum black tube maintained at a temperature of 600° F. where the SO_3 and oxygen of the air combine with the production of SO_2. The gas now passes to a treating tank containing the oil at 150° F., where the SO_2 decomposes evolving nascent oxygen which oxidises the sulphur compounds with the formation of SO_2. As a result of the treatment precipitates are formed in the treating chamber, while the gases consisting of SO_2, air, and unconverted SO_3 pass to a scrubbing tower containing concentrated sulphuric acid where the SO_3 is absorbed. The remaining SO_2 and air pass to charcoal absorber where the sulphur dioxide is absorbed, the air being allowed to escape through an air vent. When the treatment has reached the desired point the flow of oxidising gas is stopped, and the treater is then flushed out with a diluent gas which has been previously dehydrated. The precipitates produced during the treatment are removed from the pure oil either by means of a filter press or by centrifuging.

It is essential, for the process to be carried out effectively, that both the oil and oxidising gas used should be absolutely dry, otherwise water is formed which dissolves the SO_2 producing sulphuric acid. The sulphuric acid then reacts with the oil forming acid tars, thus causing a considerable loss during the treatment.

Examples of the action of nascent oxygen on sulphur compounds occurring in various petroleums are quoted.

The main object of the process is to provide an economical method for producing pure stable products without acid sludge losses, and for the formation of by-products of commercial value. H. E. T.

496. Improved Process for the Production of Ethylene. J. W. Johnson. E.P. 294,787. Aug. 27, 1928.

Ethylene is produced by the interaction of acetylene and hydrogen by passing the gas mixture in a finely distributed condition into an inert liquid (tetrahydro- or decahydronaphthalene) at a temperature not less than 100° C., the solvent having in suspension the catalyst (nickel or, better, palladium) deposited on a carrier (kieselguhr). Pressure is applied if desired. Especially when palladium is used, an excess of hydrogen may be employed with very little formation of ethane. W. S. N.

497. Production of Gaseous Hydrocarbons from Gas Mixtures Containing Hydrogen and Oxides of Carbon. J. Y. Johnson. E.P. 293,572. July 13, 1928.

Gaseous olefines (ethylene, propylene and butylene), together with ethane and propane, and not more than traces of liquid hydrocarbons, are produced by passing mixtures of hydrogen and oxides of carbon (*e.g.*, water gas) at ordinary or elevated pressures and preferably below red heat (*e.g.*, 100–400° C.) over catalysts composed of iron or cobalt in conjunction with a noble metal or with a difficulty reducible metallic oxide at a substantially lower velocity than that required for the production of liquid hydrocarbons or their derivatives. W. S. N.

498. Production of Alcohol and Acetone from Vegetable Matter. A. C. Thaysen, L. D. Galloway and W. E. Bakes. E.P. 293,514. April 30, 1928.

Straw, grass and other vegetable residues and tissues containing hemicelluloses or other polysaccharides hydrolysible by dilute acids to yield pentoses, are first treated with dilute acid. The material is then drained or pressed and steamed at atmospheric pressure, care being taken to exclude infection from outside sources. The soluble carbohydrates are removed by extraction with sterilised water, the mash is neutralised by the addition of alkali and fermented by pentose-fermenting types of micro-organisms such as *bacillus acetoethylicus* (Northrop), the volatile products being collected by distillation. W. S. N.

Crude Oil.

499. Lion Plant Extracts Special Products from Smackover Crude. G. Reid. *Refiner*, 1928, 7, 7.

The products mentioned are obtained from a plant erected in 1922 of 12,000 barrels capacity, manufacturing products down to road fluxes.

The plant consists of shell stills for handling the crude, units of the Burton cracking process and stills equipped with bubble type of fractionating column.

The crude is first dehydrated by passing it through two heaters held at 200° F. and is then returned to storage for settling. A large percentage of water, salts, and sediment thus settle from the oil, after which the crude is passed through two retorts and in its passage through the tubes is raised in temperature to 300° F. On leaving the last tubular retort the crude is conducted to the top of a large flash chamber, where it meets a stream of

superheated exhaust steam. The light oil and water are thus distilled off and pass to a condenser and storage. The remainder of the crude settling to the bottom of the chamber separates its water and foreign matter and is then pumped to the stills, which are fitted with Pyrex glass gauges and automatic check valves. A. R. S.

500. Cracking Topped West Texas Crude Oil. W. F. Farager and J. C. Morrell. *Oil and Gas J.*, 1928, 27, 9.

Crude oils from various pools of the West Texas oil fields were investigated and it appears that cracking is the best way to utilize the topped crudes.

Distillation and sulphur analysis show that these oils vary considerably in their composition. Four of the eight samples show the presence of magnesium and calcium chlorides. These will produce hydrochloric acid and corrode fractionating apparatus and condensers. All the oils produce hydrogen sulphide when heated under pressure in varying degrees in proportion to the sulphur content of the crude oil.

For convenience, the N.E.P. naphtha was removed by distillation in a batch still using fire and steam and the topped crude was used as charging stock for the cracking still. Four of the oils, representative of the production of the West Texas fields, were cracked by the non-residuum process for the production of maximum yield of N.E.P. gasoline. The yields obtainable are practically the same in all cases approximating 60 per cent. with 15 per cent. pressure distillate bottoms, which on further cracking yield a further 7·5 per cent., making a total 67·5 per cent. N.E.P. gasoline. Thus by topping and cracking a final N.E.P. product of 75 per cent. of the crude oils is possible, and the blend of the straight run with the cracked gasoline in the proportions obtained from the crude oil requires very little casing head spirit to adjust the boiling points to meet the Navy specification.

By the non-residuum process, 45 lbs. of coke and 480 cu. ft. of gas per barrel of raw stock were made whilst by the residuum process, 25 lbs. coke and 290 cu. ft. of gas per barrel were produced.

The gasoline prepared from the Texas oils shows a higher anti-knock value than the average Mid-Continental gasoline. R. P.

501. Crude Oil Preheater. W. L. Gomory. U.S.P. 1,670,805. May 22, 1928.

The improved preheater comprises a vertical cylinder of sheet steel. At the top is a dome with vapour pipe to convey vapours to a condenser.

The crude petroleum is introduced into a central column, having an enlarged bottom part, and moving slowly downwards absorbs heat from the surrounding body of oil. It is then slowly discharged from the bottom of the column and evenly distributed near the bottom of the outer cylinder. The temperature of the oil increases as it floats slowly upwards on the following body of cooler oil, which is continually supplied to the bottom of the tank. A large part of the water contained in the oil separates at the bottom of the central column and may be withdrawn periodically.

The petroleum is heated by means of steam coils or coils containing the hot products of distillation. R. P.

502. Treating and Refining Petroleum. A. M. Nastukoff. E.P. 289,920. Jan. 28, 1927.

The process claimed consists of subjecting crude oil to the action of formaldehyde in the presence of strong sulphuric acid, four volumes of acid and one volume of 40 per cent. solution of formaldehyde to one volume of oil being suggested, a hardened product being formed. The mixture is

heated, diluted with water and treated with steam for the removal of light hydrocarbons. The hardened product is filtered, neutralised and washed and the absorbed heavier hydrocarbons removed by cold solvents. The filtered product may be directly cracked for illuminating oil production.

G. S.

503. Treatment of Crude Oil. J. C. Umbarger. U.S.P. 1,675,775. July 3, 1928.

An apparatus for the removal of sediment and water from crude oil consists of a vertical tank having an outlet near the top and an oil inlet near the bottom, means for maintaining the water level and means between the water level and the oil inlet for heating and causing the water to move circumferentially to the tank. The heating and circulating may be done with a steam coil having its outlet directed circumferentially to the tank. A second steam coil may be provided above the water level, and a third steam coil may be provided within the inlet to heat the incoming oil. The discharge from the third coil may be directed into the first or second coils. G. S.

504. (I.) Process for. (II.) Apparatus for Fractional Extraction of Petroleum Hydrocarbons with Alcohol. T. A. Werkenthin. (I.) U.S.P. 1,680,352; (II.) U.S.P. 1,680,353. Aug. 14, 1928.

(I.) Cracking stock, lubricating oils of different grades and residuum are produced from topped crude oil by successive extractions, using anhydrous alcohol at a temperature varying between 150° and 175° F.

(II.) The apparatus consists of a number of substantially similar units. Each unit comprises an extractor tank, a settling tank, a still for removing alcohol from the alcoholic extract, an alcohol condenser and a combined storage tank and alcohol dehydrator in which a dehydrating material is suspended, also a safety condenser attached to the extractor tank, and means for introducing oil and alcohol and for steam-heating the extractor tank and still. W. S. N.

See also Abstract No. 530.

Natural Gas.

505. Flow of Gases through Small Orifices. E. L. Rawlins. *Oil and Gas J.*, 1928, **26**, 25.

This paper is a forerunner of one to be published by the Bureau of Mines on the subject of gas flow through orifices when the pressure drop is above the critical. In this case the velocity in the orifice has risen to a definite maximum value, and the flow then depends upon the upstream pressure only. It is not common flow-meter practice to employ a pressure ratio above the critical, and normally the volume of gas flowing through the orifice will depend upon both the up-stream and down-stream pressures.

The method of construction of the rather small diameter orifices used is given, together with a brief survey of the apparatus and means of carrying out the determination.

Graphs are given showing the relation between the value of the coefficient C in the discharge formula $Q = \frac{CA\sqrt{P_1(P_1 - P_2)}}{\sqrt{T}}$ (where P_1 = upstream pressure and P_2 = downstream pressure) and the ratio P_2/P_1 is given for straight and trumpet shaped orifices. Owing to the variation in the values of the ratios of the specific heats for air and natural gas, it is necessary to give separate graphs in the above cases for each gas.

The formula for use when the flow is critical is given, and the figures obtained for the two types of orifice on air and natural gas are quoted. P. D.

506. The Solubility of Gases in Liquids. F. E. E. German. *Oil and Gas J.*, 1928, **27**, 4.

The properties of large surfaces of altering the concentration of a solution by adsorption are well known in the realm of physical chemistry. If this knowledge is applied to the solution of natural gas in oil in the presence of small sand grains, any theoretical conclusions we may arrive at cannot be tested owing to conflicting experimental results.

Theory indicates that for sand grains of the size normally encountered, the increase in solubility (if the adsorption is positive) will be extremely small, the extra volume of gas dissolved in the oil measured at N.T.P. being about 10 per cent. of the volume of the solid sand. The effect of curvature of the sand surface would be to give greater adsorption if the sand was dispersed in the oil since the adsorbing surface would then be convex, whereas if the sand is packed the reverse is the case.

Experimental work has not shown in the case of gas dissolved in oil in the presence of sand whether the adsorption is positive or negative. The author considers that the reason for the conflicting results which have been obtained may be found in the influence the sand has upon the rate of solution as opposed to the quantity dissolved. The provision of a large surface may have the effect of bringing about equilibrium very rapidly; whereas in the case of oil uncontaminated with sand, the rate of solution depends solely on the diffusion of the gas from the surface layer. The attainment of equilibrium would thus be delayed and unless this fact is taken into account when making observations a false result that gas dissolves more freely in the presence of sand may be obtained.

If this view of the more rapid attainment of equilibrium is correct, it would not be necessary to conserve the dissolved gas in an oil sand, since if gas is available for injection it would be possible to repressure the oil quite rapidly when desired. P. D.

507. The Reid Vapour Pressure Test. *The Oil and Gas J.*, 1928, **27**, 15.

After considering various methods of estimating the vapour pressure of natural gasoline the Natural Gasoline Association of America has decided to recommend the adoption of the method described by Reid as that best combining accuracy with ease of working.

The apparatus consists of a bomb and a water bath, which must be kept at 90° F. The bomb consists of a standard pressure gauge reading up to 45 lbs., a central or air bomb, and a lower or gasoline bomb. The latter is about ¼ the capacity of the air bomb. All three fit together and must be leak proof.

The gasoline bomb is filled with liquid which must be poured in cold. The air bomb (from which all hydrocarbon vapours have been purged by means of hot water) and gauge are screwed into position. The bomb is placed in the bath, removed, shaken and again replaced until a steady gauge reading is obtained. After suitable correction has been made for the increase of the pressure of the air above atmospheric, owing to heating up to the bath temperature, the gauge reading is taken as the vapour pressure of the natural gasoline. P. D.

508. Means of Effecting an Intimate Contact between Gases and Liquids which have to React upon Each Other. H. Petersen. E.P. 295,856, Oct. 10, 1927.

The reaction chamber is provided with a propeller which extends substantially across the entire section of the enclosure and is so positioned near one wall that it imparts a strong continuous whirling motion to the whole of the gases contained in the reaction space, thereby increasing the speed of the gases and length of their path without causing admission or removal of the gases. Secured to the propeller blade, so as to rotate therewith, is a device for dispersing the liquid having the shape of a frustum of a pyramid, with stepped profile, the larger diameter being situated next to the propeller blade, and having side openings through which liquid can be ejected. H. S. G.

509. Apparatus for Effecting Intimate Contact of Gases and Liquids. Meldrums, Ltd., and F. Marsden. E.P. 294,680, April 28, 1927.

The liquid is broken up into a fine spray by means of a rotary atomising device comprising a hollow inverted truncated cone disposed vertically within the apparatus and having internal spirally arranged vanes adapted to raise the liquid, in which the lower part of the cone is submerged up to the upper end thereof, where the wall is provided with preforations above the liquid level. The upper portion of the atomising device is closed, domed or turned inwards towards its axis to prevent the raised liquid to pass out, except through the perforations. H. S. G.

510. Improvements Relating to Absorption Processes and Apparatus. S. Pilat. E.P. 295,874, Nov. 22, 1927.

Gases are passed through layers of carbon first one way and then in the opposite direction, in such a manner that the gases are led in the first stage of the absorption process through the whole height of layers of absorption means, whilst in the second stage gases are led only through part of the layers, in containers suitably sub-divided. The process may also be carried out in the reverse direction, and by the provision of a plurality of containers it is possible to carry out the changing of the direction of flow as a continuous working operation. H. S. G.

See also Abstracts Nos. 488 and 540.

Motor Spirit.

511. Sulphur Toleration in Gasoline. G. Egloff and C. D. Lowry. *Oil and Gas J.*, 1908, **27**, 12.

There is constant pressure on the oil industry to limit the sulphur content of gasoline to 0·1 per cent. lest automobile engines be injured by corrosion.

It has, however, been shown that fuels of much higher sulphur content, even carbon disulphide, can be used provided that water is eliminated from the crank case. When the crank case oil does contain emulsified water there is said to be no corrosion when the air temperature is above 40° F., and even when it is colder than this corrosion is not marked where the engine is in regular daily use.

It has, therefore, been suggested that in summer refinery costs could be considerably cut down without detrimental effects on the engine, the old sulphur toleration of 0·1 per cent. being adhered to in winter. A. R. S.

512. Sources of Increased Gasoline Supply. C. O. Willson. *Oil and Gas J.*, 1928, 27, 11.

The Bureau of Mines report for the period ending May 31 shows that during the first five months of 1928 gasoline production was 9,997,000 barrels more than the production for the corresponding period of last year. This increased production is divided as follows: 37·4 per cent. straight run from crude oil, 49·8 per cent. by cracking of gas and fuel oils and 12·8 per cent. from casing head gas.

The importance of cracked gasoline has varied during the past three and a half years. Cracking was a minor factor until 1922-23, when it began to increase. In 1925 a rapid expansion took place and the output of cracked gasoline in 1926 showed an increase of 25 millions barrels above the 1925 output. In 1926 cracked gasoline accounted for 67·1 per cent. of the increased output of gasoline, but high prices contributed to the high output of that year. In 1927 a declining market brought a slump in the installation of new cracking units, which is shown in the low increase in production—7½ million barrels only—slightly less than the increase in straight run gasoline. In that year, however, the increase in casing head gasoline balanced the difference.

The change in market conditions, *i.e.*, declining fuel and gas oil market in 1928, is responsible for the increase in cracking activity, resulting in a production of almost 50 per cent. of the increase, and has made cracking operations more profitable.

Just what the desirable ratio between crude oil and cracking capacity should be will vary with the conditions, but in certain areas the saturation point in cracking facilities has apparently not been reached for the normal increase in gasoline demand. Natural gasoline is more than holding its own as a source of motor fuel, for the output has increased to 11 per cent. of the total gasoline production during the last 15 years. With an increase of 6½ million barrels in 1927 the position of natural gasoline was strengthened, representing 28½ per cent. of the total increase in production. In 1928 the rate of increase has fallen, although still showing 8 per cent. increase over last year, which means that the rate of increase still remains greater than the rate of increase of straight run gasoline. It is more difficult to gauge the future of natural gasoline than either crude oil or cracked gasoline, as the former depends entirely on new fields and crude oil developments. R. P.

513. Clay Treatment of Refinery Gasoline. C. O. Willson. *Oil and Gas J.*, 1928, 27, 13.

Gasoline obtained by cracking in Cross units is first washed with caustic soda solution, it is freed from the soda in settling tanks and is then mixed with a mixture, 50 per cent. acid tar, 50 per cent. fresh sulphuric acid, 6 gallons of mixed acid per barrel of gasoline. The mixture is passed through a centrifugal pump thence to a centrifuge, where the acid tar is separated. The gasoline is then agitated with 3 lbs. fine clay (200 mesh) per barrel; large paddles are used for agitation; it is finally freed from the clay in a Sweetland filter press. The resulting gasoline is found to be completely stable as regards colour. W. N. H.

514. Continuous Treating of Gasoline. G. T. Yost. *Oil and Gas J.*, 27, 14, 120.

The following scheme for refining pressure distillate has been adopted as standard by the Pure Oil Company. The untreated oil is fed to a centrifugal pump, to which a small acid pump delivers sulphuric acid and which forces the oil and the acid through an orifice mixture, thence to an acid settling

tower from the top of which oil passes to a second acid settling tower, sludge being withdrawn from the base of each tower. The oil is then similarly treated by means of orifice mixers and settling towers with water and caustic soda solution. The inlet to each of the six settling towers is located near the base, and is extended vertically upwards, the end being plugged and discharge into the body of the tower is effected by means of five perforated side-lines, which give a spraying effect. To obtain efficient neutralisation of traces of suspended acid, an excess of soda is used, means being provided for returning to the soda orifice mixer a part or all of the lye from the soda separating towers. W. S. N.

515. Improved Treatment of Gasoline and Similar Petroleum Products. A. G. Bloxam. E.P. 295,253, Aug. 7, 1928.

The improvement in the method of treating gasoline and similar petroleum products consists in the elimination of saturated hydrocarbons of high boiling point. The gasoline is fractionated into two parts, to obtain a higher boiling part and a lower boiling part, the cutting temperature being 160° C. to 170° C. The unsaturated hydrocarbons in the higher boiling point fraction are then extracted by means of liquid sulphur dioxide or acetone; and after removal of the solvent are added to the fracture boiling up to 160° C.

The gasoline obtained in this manner exhibits distinct improvements over the corresponding untreated gasoline. In particular, it possesses increased volatility and the undesirable tendency to produce knocking when subjected to high compression in an internal combustion engine is greatly reduced. H. E. T.

516. Liquid Phase Purification of Distillates. T. G. Delbridge and J. B. Hill. U.S.P. 1,682,603, Aug. 28, 1928.

A method is described for producing lower from higher boiling point hydrocarbon oils and of purifying the lower boiling oils. It comprises, subjecting the higher boiling oils to cracking temperatures under super-atmospheric pressure, and fractionating the resultant vapour by contact with reflux liquid. The vapours are condensed under super-atmospheric pressure and the condensate brought into contact with fuller's earth or other catalyst to effect polymerization of gum forming and colour-imparting bodies. This is carried out at a temperature substantially above its normal boiling point, but under a pressure of at least 30 lbs. per sq. in., sufficient to maintain it in the liquid phase. By this means the polymerized products remain in solution in the oil instead of being absorbed by, and thus contam nating the catalyst. Separation of the lower boiling-point vapours from the polymers in solution is effected by distillation at materially lower pressure, utilising in the distillation heat abstracted from the untreated fractionated vapours. H. S. G.

517. Process for Refining Petroleum Oils with Doctor Solution. R. L. Hallett, assr. to National Lead Co. U.S.P. 1,678,984, July 31, 1928.

The process consists of a method of refining gasoline and similar petroleum products containing sulphur by means of a doctor solution prepared by dissolving dry lead chloride in an aqueous solution of caustic soda. The new refining solution contains sodium plumbite together with sodium chloride, and is formed according to the following equation,

$$PbCl_2 + 4\ NaOH = Na_2PbO_2 + 2\ NaCl + 2\ H_2O.$$

The presence of sodium chloride in the refining solution has no effect on the purification of the petroleum, but its presence considerably facilitates the separation of the refining solution from the petroleum. H. E. T.

518. Purification of Benzol Fractions. C. Still. E.P. 288,591, Aug. 31, 1927.

Harmful and corrosive constituents of commercial benzol fractions are entirely removed by treating them, immediately on production, with an aqueous caustic alkali solution. The process is advantageously and conveniently conducted in a plant consisting in a distillation and rectification column supplied with steam, a dephlegmator above the column, and a condenser attached to a receiver and separator. The mixture of light oil and water is separated and the light oil passed continuously into a pipe into which an 18 per cent. caustic soda solution is introduced, the proportion of light oil to caustic soda being about 30 : 1. The mixture passes through a centrifugal pump, which brings about intimate admixture, and then into a separator, from which the bottom layer of caustic solution separates continuously. H. S. G.

See also Abstracts Nos. 546, 548, 549, 606, 610 and 614.

Kerosine.

519. Process for Refining of Petroleum Fractions. F. C. Axtell. U.S.P. 1,667, 425. July 17, 1928.

A process is described for refining petroleum fractions by means of either persulphuric acid or Caro's acid in sulphuric acid solution. The persulphuric acid is prepared by dissolving either sodium or potassium persulphate in excess of concentrated sulphuric acid, and on standing it is slowly converted into Caro's acid.

The oil to be refined is given a preliminary treatment with 5 per cent. of dilute sulphuric acid, followed by soda and water washes. The treated oil is now thoroughly agitated with the persulphuric acid in sulphuric acid solution for one hour, the quantity used depending on the nature of the oil.

It is claimed that persulphuric acid in sulphuric acid solution has a distinct advantage over common oxidising agents used in refining, in that it removes sulphur compounds, nitrogenous bases and acids, without apparently attacking other valuable hydrocarbons in the oil. H. E. T.

520. Apparatus for Treating Liquids to Remove or Recover Substances Therefrom. E. B. Miller. U.S.P. 1,678,299. July 24, 1928.

A prior application (Serial No. 447,857) refers to a process of separating solutes from solutions, whilst application Serial No. 544,186 refers to the refining of oils by such a method. The present invention relates to apparatus for carrying out the process described in the above applications, which consists in removing a substance from a solution by adsorbing it in a porous material having pores of ultra-microscopic size.

For the purpose of absorbing the substances in solution from the petroleum product to be refined, the crude liquid flows through a series of agitators and separators wherein the liquid and adsorbing material such as silica gel are brought into intimate contact and then separated, the material moving in one direction and the liquid in the other. In this way a part of the objectionable constituents are removed at each stage as the liquid flows through the agitators, until finally the refining is completed in the last agitator, where the liquid comes in contact with freshly activated absorbing material.

In order to activate the adsorbent material, it is first washed with cold water, which displaces the lighter and more valuable fractions of the adsorbed liquid. A further treatment with hot water is then given which removes a smaller fraction consisting of high boiling constituents high in sulphur and

unsaturated compounds, and suitable as a flotation or fuel oil. The porous material having been freed by water displacement of the adsorbed constituents is filtered from the wash water, run into an activator, and its moisture content reduced to 6-8 per cent., rendering the gel ready for re-use. Where the efficiency of the absorbing material has decreased owing to deposition of carbonaceous or gummy materials thereon, an auxiliary activator may be employed in which volatile material is first distilled off in a current of steam or non-oxidising gas, and the temperature finally raised sufficiently to burn off the carbon in a current of air and furnace gases. H. S. G.

See also Abstract No. 517.

Lubricating Oils and Wax.

521. WHAT IS LUBRICATION? MOLECULAR FORCES IN THE MECHANISM OF LUBRICATION. R. Ascher. *Erdöl und Teer*, 1928, **4**, 424–425.

The coefficient of bearing friction, R, is given by the expression ZN/p, where Z is viscosity of the lubricant, N velocity of rotation, and p the pressure in kgm./cm.^{2} between the moving metal surfaces. Normally R varies gradually and continuously, but should the oil film break, R will increase suddenly. The tendency to break is determined by the cohesion and adhesion of the oil. An estimation of adhesive power may be obtained by measuring the angle of contact of the oil with a metal surface. If this is less than 35° the oil will have good lubricating properties. The angle of contact may be lowered by the addition of soaps, oils containing unsaturated groups, or flowers of sulphur. Amongst the factors enumerated which should govern the choice of a lubricating oil, high speeds and low pressures require oils of low viscosity, whereas for low speeds and high pressures high viscosity oils are more suitable. W. S. N.

522. LUBRICATING OIL CHARACTERISTICS OF MINERAL OILS AND TAR OILS AND THE INFLUENCE THEREON OF THEIR CHEMICAL COMPOSITION. J. Herzenberg and E. Jungfer. *Erdöl und Teer*, 1928, **4** (I.) 370–373, (II.) 387–390, (III.) 406–408.

A machine oil of the Deutschen Erdöl A.-G. at Rositz, cold-produced from brown-coal generator tar, is found to contain 24 per cent. of phenols, 0·6 per cent. of hard asphalt, about 15 per cent. of resins, and 60 per cent. of neutral oil, the last two being separated by distillation. The neutral oil contains 10–12 per cent. aromatic hydrocarbons, 2 per cent. hydroaromatic hydrocarbons, 20–25 per cent. saturated hydrocarbons, 50–60 per cent. unsaturated hydrocarbons, and 8–10 per cent. derivatives of oxygen and sulphur. The greater part of the labile unsaturated constituents and of the oxygen compounds of the neutral oil is removed by distilling thrice over sodium. Aromatic hydrocarbons are separated as their picrates from the distillate, and are recovered by warming with ammonia. The non-aromatic residual neutral oil is dehydrogenated at 380° over nickel carried on alumina and the resulting aromatic hydrocarbons removed with picric acid. Saturated hydrocarbons are obtained from the neutral oil, after removal of the original aromatics, by repeated treatments with sulphuric acid of increasing concentration (50 per cent. to 98 per cent.). The unsaturated resins contained in the original machine oil are 64 per cent. distillable at 0·5–0·6 mm.; the steep viscosity-temperature curves of the distillates indicate that

the resins consist chiefly of condensation products of cyclic unsaturated hydrocarbons. Chrysene is isolated from the product of destructive distillation of the resin. Specific gravity gives an indication of the source (coal tar or petroleum) of a lubricating oil. The specific gravity of the neutral oil here dealt with is influenced by the resins rather than by the smaller quantity of aromatics. Saturated hydroaromatic and, in part, unsaturated hydrocarbons have a lower flash point than aromatic and other lubricating oil constituents. The very low setting point, —20°, of the aromatic hydrocarbons recovered from the picrates indicates the presence of side chains; the relatively low setting point, —1·5°, of the saturated hydrocarbons and the high specific gravity indicate the presence of cyclic compounds. Ease of evaporation is increased by removal of resins, but is lower again in the aromatic and saturated hydrocarbons; thus, apart from the resinous bodies, the unsaturated hydrocarbons are appreciably more volatile than the aromatic and saturated hydrocarbons. High tar-formation numbers usually prohibit the use of brown-coal tar oils as transformer or switch oils. At corresponding b.p. ranges the unsaturated constituents of brown-coal tar lubricating oil have greater viscosities than the saturated constituents; this again indicates that the unsaturated hydrocarbons have a cyclic structure. The viscosity of brown-coal tar oil is raised by removing the paraffins, and lowered again by removing the phenols. The viscosity of decahydro- is greater than that of tetrahydronaphthalene; in this series viscosity is also increased by methylation. To characterise the influence of temperature on the viscosity of an oil it is proposed to use the mean decrease in viscosity for a 10° C. temperature increase, taken preferably from a series of such 10° C. intervals within the temperature range of the machine in which the oil is to be used.

W. S. N.

523. Decolorising Agents. Büttner-Werke A.G. and F. Kleinmann. E.P. 295,623. August 14, 1928.

This invention relates to the production of decolorising agents of high efficiency, e.g. :—

1. By heating a mixture of dried ground clay and carbon produced by the imperfect combustion of hydrocarbon oils and cooling gradually.

2. By heating a mixture of dried ground peat with quicklime, heating the resulting product with acids, and mixing the residue with fullers earth, hydrated aluminium silicate, kieselguhr, etc., and heating again.

3. The residue produced by mixing heated dried ground clay with petroleum residues and heating until the oil is burned off.

4. By mixing oil-free carbon black with ordinary filtering material such as china clay, and by subjecting a mixture of kieselguhr and starch to dry distillation.

Substances of excellent decolorising properties may be prepared, if care is taken that an intimate mixture of fullers earth and carbon is produced by mixing in the presence of liquids having good wetting properties. As starting materials fullers earth, hydrated aluminium silicates, such as Florida earth, floridin, etc., and carbon such as soot or American lustrous soot obtained from American natural gas may be employed. The mutual combination of the particles is effected by grinding in the presence of a liquid such as water, alcohol, acetone and carbon tetrachloride. The mutual combination of the particles may also be furthered by binding agents, such as small quantities of silicic acid gel and colloidal hydrates of alumina. The decolorising agent so produced is filtered and dried in order to remove the wetting agents, before being used as a decolorising substance.

H. E. T.

524. Improvements in or Relating to Lubricating or Like Oils. H. L. Callendar, R. O. King, and J. Mardles. E.P. 295,230. August 7, 1928.

Lubricating oils when used in parts of machinery subjected to heavy loads and high temperatures, undergo partial oxidation, with the formation of highly-complex and unstable organic peroxides. These organic peroxides readily decompose giving rise to tarry, resinous and asphaltic residues. To prevent this partial oxidation of the oil with the consequent formation of gummy residues, lead tetra-ethyl and ethylene dibromide are added to the oil, thus producing a compounded lubricating oil which is heat resisting and has little or no tendency to undergo partial oxidation. H. E. T.

525. Continuous Distillation of Hydrocarbon Oils. R. D. Hunneman. U.S.P. 1,676,611. July 10, 1928.

A process particularly adapted for the production of viscous residues and distillates, is described. Oil to be distilled is fed with steam to a pipestill, whence, heated to the desired temperature, it passes through an enlarged equilibrium conduit to a chamber of substantial cross-sectional area so that the velocity of vapours passing through is materially lowered. From the separating chamber unvaporized liquids pass through a trap and discharge into the upper portion of a column equipped with suitable fractionating plates. Steam supplied to the bottom of this stripping column passes counter-current to the descending liquid carrying off vapours through the top of the column, and into a line leading to an intermediate point of the coil in the pipe still. From the separating chamber vapours pass to a fractionating column where fractional condensation takes place, the condensate being withdrawn from the base of the column. The uncondensed oil and steam pass through a vapour line to a condenser. In the production of lubricating oils a high vacuum is maintained on the apparatus. H. S. G.

526. Distilling Hydrocarbon Oils. R. D. Hunneman. U.S.P. 1,676,609. July 10, 1928.

This is an improved method of distillation, more particularly of lubricating oils for the removal of lighter fractions, and the production of viscous lubricant residues. The oil to be distilled is forced by a pump through a heating coil set in a furnace. Steam may be supplied at an intermediate point. From the coil the heated oil passes into an enlarged chamber in which a separation of vapours and unvaporized liquid takes place. The liquid descends in the column, through stripping plates, counter-current to steam admitted to the column through a distributor at or near its base. Liberated vapours and steam rise through fractionating plates, the condensate refluxing to the lower portion of the column. Additional or controlled cooling of the vapour, before issuing from the column, is effected by means of a heat exchange coil. Vapours and steam pass through the vapour line to a condenser or a trap may be inserted at an intermediate point in the condenser, and steam and uncondensed vapours withdrawn by a pump and forced back into the oil-supply line. A vacuum may be maintained on the tower and heating system, an absolute pressure of 10 to 50 mm. being desirable where the oil enters the tower. H. S. G.

527. Process for Refining Oils and Waxes. W. A. Patrick and E. B. Miller. U.S.P. 1,678,298. July 24, 1928.

U.S.P. 1,537,260 refers to the discovery that porous bodies having ultra-microscopic pores will absorb certain solutes from solution. The present invention relates to the adaption of this process to the refining of petroleum products.

The petroleum product is refined by bringing the crude product into intimate contact with an absorbing material such as silica gel, allowing the two to remain in contact long enough for the objectionable impurities to be adsorbed and thereafter removing the refined product from the material. The adsorption may be carried out by percolation through a tower filled with adsorbent material, the refined product being drawn off from the bottom of the tower. After the gel has become saturated with impurities, the crude is diverted to a second similar tower, whilst the gel in the first tower is activated by suitable means. A preferred method however consists in causing a stream of crude liquid to enter at one end of and to flow successively through a series of agitators and separators containing silica gel which is fed in continuously at the opposite end to that at which the liquid enters and by appropriate means made to move in a direction counter-current to the liquid flow.

Liberation of the adsorbed impurities from the adsorbing material may be accomplished by driving out these materials by immersion in a liquid such as water, and then activating the material by driving off or liberating the material. For products having higher boiling points, steam activation may be employed in addition. Where the adsorbing efficiency has decreased owing to contamination with carbonaceous or gummy materials, an auxiliary activator may be employed in which the material is heated in a current of hot gases containing just sufficient oxygen to burn off the carbonaceous matter. H. S. G.

528. Improvements in or Relating to the Regeneration or Purification of Lubricating Oils. Swan, Hunter and Wigham, Ltd., and H. J. Young. E.P. 292,300. 1928.

A method is described for the purification of lubricating oils which, after use in engines are found to corrode steel and white bearing metals. The corrosion is caused by the presence in the used oil of free sulphuric acid and metallic sulphates, the chemical estimation of which gives no guide as to the corrosive nature of the oil. Purification is effected by bringing the oil, preferably warm, into intimate contact with a non-ferrous metal, such as zinc, which reacts with the corrosive impurities to produce products which are non-corrosive. The purifying agent is arranged in the form of hollow or perforated plates through which the oil is continually circulated at about 70° F. H. E. T.

529. Distilling Hydrocarbon Oils. R. E. Wilson. U.S.P. 1,676,610. July 10, 1928.

The process is particularly adapted to the production of viscous lubricating oil from reduced crude oils or from distillates. Oil to be distilled is pumped through a pipe coil in which it is heated to the desired temperature. Steam and hydrocarbon vapours are forced into the heated oil passing through the coil. The heated oil and steam pass from the coil into an enlarged conduit and into a vaporizing drum. The enlargement of the conduit permits a substantial attainment of equilibrium between the heated oil and vapours at the minimum pressure prevailing in the system. In the drum, separation of vapour and liquid takes place, vapours passing off to a condenser, and the resulting condensate to a receiver. Unvaporized oil passes through a trapped line into a tower where it descends counter-current to rising steam from a perforated distribution pipe. The steam completes the removal of light fractions from the oil, leaving as a residue the desired viscous lubricating fractions substantially cooled by their contact with the steam. Steam and vapours carried off pass through a pipe and are admitted into the stream of

heated oil before it enters the enlarged conduit. If desired the operation may be conducted under vacuum. H. S. G.

See also Abstracts Nos. 504 and 532.

Special Products.

530. Making Asphalt at New Orleans Plant. N. Williams. *Oil and Gas J.*, 1928, 27, 13.

Mexican and Venezuelan crudes giving about 65 per cent. asphalt are generally used, although West Texan crude are used when the asphalt demand is low on account of the higher spirit yields obtained.

The crude is pumped through preheaters where the asphalt from the stills is used, counter-current, into Trumble pipe stills. After passing through these the oil passes into vaporizer towers at the tops of the stills. These towers are constructed of two concentric sheils, the outer of which is brick insulated and which conducts flue gases at definite temperatures, depending on the desired consistency of finished asphalt, while the inner column serves to separate and fractionate the crude. The overhead distillate passes through dephlegmators, while the asphalt passes to storage via the preheaters.

Oxidized asphalt is prepared from a light asphalt by blowing with compressed air in a heated vessel.

Dubbs coke is used as pulverized fuel in the fire boxes and it is claimed that more complete combustion than is general is obtained by using a pilot flame burning gas obtained from the Dubbs unit in use in the refinery. A. R. S.

531. Improvements in Cold Bituminous Paving Composition. O. H. Berger. E.P. 293,830. July 2, 1928.

This is a modification of Spec. 257,114, September 28, 1925, relating to the making of a cold-lay paving mixture, which comprises adding to the mixture of asphalt cement and mineral aggregate after the admixing thereof 3–5 per cent. of an ameliorating agent which may be water and/or fresh oil, and mixing until a light brown colour is obtained. Such addition acts to prevent any adhesion of the particles of the original mix, thus ensuring a long retention of life and prevention of any lumping which might otherwise occur.

H. S. G.

532. Refining Oils. L. Burgess. U.S.P. 1,681,895. August 21, 1928.

The process relates to the production of white lubricating oils. The oil is treated with successive portions of 10 per cent. to 15 per cent. of fuming sulphuric acid, the sludge settled off in each case, after which it is neutralized with caustic soda and treated with a monohydric alcohol and water in equal proportions. A. R. S.

533. Process of Working up Acid Resins Obtained from the Refining of Mineral Oil Derivatives into Neutral Bitumens. C. Sautermeister and F. Wilhelm. U.S.P. 1,667,731. July 17, 1928.

Acid resins obtained by refining mineral oil derivatives are worked up into neutral bitumens by reducing the sulphuric acid contained in the acid tar by means of sulphuretted hydrogen. The latter may be added direct or produced in the bitumen by the addition of a small quantity of sulphur. The reaction occuring may be represented by the equation

$$H_2SO_4 + 3\,H_2S = 2\,S_2 + 4\,H_2O,$$

the sulphur thus formed reacting to harden the hydrocarbons in the acid resin.

In practice the process is carried out as follows:

The raw acid tars are placed in agitators and extracted by heating with steam or by direct firing. During this operation part of the sulphuric acid is separated and removed, while the acid remaining in the tar is reduced to sulphur which exerts a hardening influence on the hydrocarbons present. By estimating the acid remaining in the tar after extraction and by adding a petroleum residue of higher boiling range, the hardness and ductility of the bitumen it is desired to produce may be controlled at will. The tar is now heated to 130° C. at which point sulphuretted hydrogen is introduced into the hot mass, or a small quantity of sulphur may be added. The sulphur so added reacts with the sulphuric acid present forming sulphuretted hydrogen, which in its turn reduces the acid to sulphur and water. This cyclic process continues until all the acid has been reduced by the sulphuretted hydrogen, the end of the reacting occurring when frothing ceases. The bitumen thus obtained is very shiny and possesses great ductility. H. E. T.

534. Bituminous Coating Compositions. G. A. Wellings and E. Johnstone. E.P. 289,737. November 1, 1926.

A suitable composition for treating road surfaces, etc., is made by preparing a solution of bitumen in a solvent (petrol or benzol) and adding thereto sulphur in solution or suspension in a liquid miscible with the bitumen solvent. The sulphur is preferably added as a saturated solution in carbon bisulphide. For hardening purposes, gold size may be added, preferably prior to the addition of the sulphur. G. S.

See also Abstracts Nos. 499 and 502.

Analysis and Testing.

See Abstracts Nos. 507, 521 and 604.

Refinery Plant.

535. Distillation. A. E. Becker. U.S.P. 1,676,724. August 10, 1928.

An improvement in the art of distillation is claimed in which the still and tower above the still are filled with metallic wool. This affords an equalizing heat transfer medium without substantially diminishing the still capacity. Furthermore, the wool in the vapour space above the still catches entrained droplets and mist, and affords a progressively graduated temperature path up the tower affording favourable conditions for good fractionation. H. S. G.

536. Temperature Controller for Oil-Separating Towers. R. H. Carr and C. B. Watson. U.S.P. 1,678,328. August 24, 1928.

The invention relates to a separating tower provided with a refluxing coil and automatic temperature controlling mechanism. The tower, of the type used in oil refining apparatus for operating various fractions of oil stock after heat treatment in a still, is constructed of a vertical cylindrical shell in which is provided internal staggered baffle walls. Heated oil vapours circulate through the tower, the more volatile fractions being carried off from the top of the tower whilst the higher boiling point fractions condense and are removed from the bottom of the tower.

To facilitate condensation and as a means of preheating the primary oil stock, the tower is provided in its upper part with one or more refluxing coils communicating with external inlet and outlet pipes. Within the top of the

tower adjacent the refluxing coils is a drum to contain water, provided with an inlet and outlet controlled by valves exterior to the tower. Entering and communicating with the bottom of the drum is a water-filled pipe-line, the lower end of which enters a diaphram chamber provided with an automatic control valve situated in a shunt pipe connecting the inlet and outlet pipes in such a manner as to short-circuit the refluxing coils.

In operation the drum is partly filled with water and also the pipe-line leading to the regulator valve. Assuming it is desired to maintain a temperature of 325° F. in the tower, the construction of the apparatus is such that at this temperature the steam pressure in the drum will be about 96 lbs. absolute, this and the hydrostatic head of water in the pipe-line being directed on the diaphragm in opposition to the forces of a lever and weight and maintaining the regulating valve open, allowing oil to by-pass before reaching the reflux coil. With increase in temperature, the steam pressure overcomes the action of the weight and the movement of the diaphram closes the by-pass and allows circulation of the primary oil stock through the reflux coils. H. S. G.

537. Improved Method and Apparatus of the Bubble Type for Treating Gases and Vapours with Liquids. F. B. Dehn for C. F. Braun. E.P. 291,527. 1928.

This apparatus consists of a tower fitted with a number of plates supporting vertical down-take pipes and bubble caps. The arrangement of down-take pipes and caps is staggered, so that the down-take from the plate above passes directly through a liquid seal into the bubble cap of the plate below. The seal is arranged in such a way that the upward ascending vapours in each cap are deflected to meet a thin layer of liquid passing from the seal down the walls of the cap. P. D.

538. Improvements Relating to the Cooling of Air and Gases for the Purpose of Condensing Vapours Therefrom. M. Fränkel. E.P. 294,354. June 22, 1928.

A process and apparatus are described for the condensation of vapours from air and gases by cooling, characterised in that one cold storing device is arranged in front of and another behind the ammonia vaporizer of a refrigerating machine, these being adapted to be changed over alternatively so that each in turn takes up cold from the dried air and then returns the cold to the moist fresh air which is to be rendered free of moisture.

In one example of the process, air which is to be deprived of moisture enters through a change-over valve into a cold storing device and cools to —10° C. or less. It then passes into the first and from thence into the second half of the ammonia vaporizer, whence it passes out through a second cold storing device after yielding up its cold in this. After the first cold storing device has become heated up to atmospheric temperature, fresh air is blown through in the reverse direction in order to dry off moisture clinging to it until the cold storing device behind the ammonia vaporizer at the time being has been completely cooled down by the dried air which has been further cooled down in the ammonia vaporizer. The change over is effected by means of automatic timing devices operated electrically or by means of compressed air, and regulated so that the change over from the ammonia vaporizer to the condenser does not take place simultaneously with the change over of the cold storing devices, but always somewhat earlier. Removal of ice is effected by spraying with brine or lye in alternate operation, that half of the vaporizer adjacent to the cold storing device acting as discharge. H. S. G.

539. Vaporizer. F. A. Milliff and J. A. Milliff. U.S.P. 1,644,937. October 11, 1927.

Hot oil from a pipe still enters a cylindrical vaporizer and is projected from a nozzle tangentially into an annular pan from which it overflows through an opening into an overflow pan. Such discharge of the oil causes a rapidly rotating current in the annular basin and a uniform distribution of the liquid over the entire cross-sectional area of the vaporizer. The mixture flows over the outer wall of the overflow pan upon a distributing deck upon which it rises up through pipes until it is above the tops of other pipes down which it flows upon the top of a series of drip screens. The hot mixture spreads in a thin film over the wire in each successive screen and the lower boiling point hydrocarbons are given off as vapours which are drawn off from the space above each screen into the lower end of the tapered tube of the screen above, and pass into a vertical vapour passage way formed by the series of tubes and out through the tube directly beneath the bubble deck plate. From here the vapours pass up through the tubes already mentioned, through screens and the mass of the dephlegmating material and out of the vaporizer through a vapour line. To accelerate the vapour passage steam may be admitted. The dephlegmating unit comprises screens and a mass of steel turnings which collect and condense any vapours heavier than those desired to pass out. Beneath the lowermost screen is a steam-heated reboiler through which the residuum is drawn off. If a test shows that products desired to be vaporized are passing out with the residuum, live steam is admitted into the residuum in the reboiler through a perforated pipe. H. S. G.

540. Apparatus for Cooling or Cleaning Gases. The Power-Gas Corporation and N. E. Rambush. E.P. 294,836. December 19, 1927.

The apparatus is adapted to be employed for cooling, cleaning or otherwise treating gases or liquids or as a condenser for removing vapours from gases or as a heat interchange apparatus comprising a casing through which one medium (for example, the gas to be treated) is caused to flow. Within the casing is a plurality of aggregates or nests of transverse tubes through which flows another medium. These aggregates are arranged in juxtaposition in horizontal layers one above the other, the tubes in each layer at right angles to the tubes in the next succeeding layer, alternate layers being connected together at their ends by independent easily removable casings or covers so as to form independent tubular systems, provision being made for cutting out one or more systems while the remainder are in operation. Such a construction enables the temperature within the apparatus to be regulated within wide limits. H. S. G.

541. Apparatus for Detecting, Indicating and Recording the Presence of Inflammable Gases. H. T. Ringrose. B.P. 293,125. April 1, 1927.

Inflammable vapours or gases diffusing through a porous vessel are burnt, thus producing a vacuum proportional to the amount present. The vacuum produced actuates a collapsible diaphragm which is utilised to complete an electric circuit and light a lamp, and in addition operate a manometer which records the percentage of inflammable gases or vapours present. H. S. G.

See also Abstracts Nos. 487, 501, 503, 508, 509, 520, 525, 526 and 532.

Engineering.

542. A New High Compression Solid Injection Oil Engine. *The Engineer*. 1928, 3787, 150.

The article describes a vertical four-cylinder four-stroke cycle engine rated at 200 B.H.P. at 330 r.p.m.

The crankshaft takes compressive stresses only, the tensile loads being carried by through bolts from bedplate to cylinder heads. The bedplate is of the box type and all lubrication passages are cored to avoid pipe work.

The cylinder liners are bolted at the upper end to the cylinder heads and are free to expand downwards.

Floating gudgeon pins are used and the pistons are made in two parts to facilitate removal and replacement.

The valves are operated by means of short push rods from a camshaft mounted at cylinder level and driven from the crankshaft through skew-gears and a vertical shaft.

Separate fuel pumps driven from the camshaft are provided for each cylinder, and the engine governor controls the fuel supply by varying the time of opening of the suction valves during the later part of each delivery stroke. This gives a constant injection timing and rate of pump pressure rise for all loads. Provision is made for hand priming of each atomiser, but a single handle operating a master air valve controls starting and stopping.

The scantlings and materials will pass Lloyds Rules and the performance conforms to B.E.S.A. specification No. 213 for heavy oil engines of the airless injection type for electrical purposes.

The makers' guaranteed fuel consumptions are :—

0·42 lbs.	per B.H.P.	per hour on	full load.
0·45 lbs.	,,	,,	three-quarters load.
0·50 lbs.	,,	,,	half load.
0·55 lbs.	,,	,,	quarter load.

The lubricating oil consumption is guaranteed at 0·0003 gallons per B.H.P. hour on full load. R. S.

543. Heat Transfer for Oils and Water in Pipes. F. H. Morris and W. G. Whitman. *Ind. Eng. Chem.*, 1928, 20, 3.

A series of experiments were carried out upon three petroleum products and water in a heat exchanger consisting of a jacketed straight pipe. The heat transfer coefficient for the oil or water film only was obtained by taking the temperature of the outer side of the pipe in which the liquid was flowing by means of peened-in thermocouples; the temperature at the inner surface was then calculated from the known heat input to the oil, and the thickness of the pipe wall.

The results were interpreted by Nusselts' equation, which expressed $\frac{H\,D}{K}$ as a product of functions of $\frac{Dus}{z}$ and $\frac{cz}{K}$,

where D = inside pipe diameter.
H = film coefficient.
K = thermal conductivity of liquid.
u = mean linear velocity of liquid.
s = density of liquid.
z = viscosity of liquid.
c = specific heat of liquid.

The form of the functions are determined for the temperature range of the experiments by graphical approximation.

Both heating and cooling runs were carried out, and it is found that if the viscosity and other properties of the fluid are taken as those at the mean temperature (and not at the film temperature) the value for the coefficient is 25 per cent. lower for cooling than for heating conditions.

Owing to lack of data a constant value was assigned to the conductivity of oil, the effect of temperature being disregarded.

The experimental figures are given in complete form. P. D.

544. Improvements in Filters for Fine Filtration of Liquids. O. Petersen. E.P. 293,668. July 12, 1928.

The filter consists of two coaxial elements mounted in a casing with inlet and outlet branches, and arranged with a clearance space between of width not greater than the finest degree of separation required. Each element is slotted or drilled with holes which may be made exactly to register.

Adjustment of one element by rotation progressively reduces the size of the free passages from the inner to the outer slots and thus regulates the fineness of separation obtainable.

The filter is designed with shallow channels between the slots to facilitate gravity settling of deposit, and the elements are closed below the filter openings to retain such deposit.

The device can be dismantled and cleaned quickly without risk of unfiltered liquid or sediment passing into the outlet pipe. R. S.

545. A Magnetic Method of Testing Welds. A. Roux. *Comptes Rendus*, 185, p. 859.

Experiments were carried out to discover whether internal defects in steel test pieces, placed between the poles of an electro-magnet, could be detected through their effect on the lines of magnetic force on the surface of the test pieces.

A U-shaped electro-magent of rectangular section, 40 mm. by 70 mm., with poles 90 mm. apart, was used. A coil of fifteen turns of wire was wound on each arm of the magnet, and a current of 70 amp`res was passed through the coils. The test pieces covered with paper were placed between the poles of the magnet, and iron filings scattered on the paper.

It was found that surfaces of internal discontinuity in the test pieces, normal to the magnetic lines of force, were shown by accumulations of iron filings at right angles to the ordinary filing lines. W. H. F.

See also Abstracts Nos. 505, 593, 594 and 596.

Anti-Detonation.

546. Engine Knock and Related Problems. A. C. Egerton. *Nature* (Supplement), 1928, 122, 3062.

A review of previous work is given. It was found that anti-knocks do not influence the position at which a detonation wave is set up when a gaseous explosive mixture is fired from the closed end of a tube closed at one end. It was found, however, that a certain increase in pressure diminished the length of flame travel before detonation set in but that further increases of pressure produced no effect. Experiments were conducted on the self-ignition temperature of petrol dropped into a heated iron pot into which air was gently blown. Anti-knocks raised the igniting temperature as follows :—

	Rise in Igniting Temp. ° C.
Bismuth diethyl	140°
Iron carbonyl	130°
Bismuth triethyl	120°
Lead ethyl	90°
Nickel carbonyl	40°
Tellurium diethyl	55°
Bismuth triphenyl	42°

Lead oxide from an arc in air was found to be ineffective, but lead vapour from an arc in argon was effective as an anti-knock to much the same extent as the same amount of lead as lead tetra-ethyl. Tested by the same method, the following metals were found to be effective as anti-knocks: thallium, potassium, lead, manganese, bismuth, selenium, tellurium, sodium cadium, calcium and antimony.

It would appear that the metallic oxide is an anti-knock if its state of subdivision is sufficiently fine. It also appears to be the common property of effective metals that a state of equilibrium exists between the oxides of the metal at the temperature of the reaction. The effect of the anti-knock appears to take place in the body of the gas rather than at the surface of the vessel.

Lead tetra-ethyl raises the igniting temperature of various substances as follows:—

Substance	Igniting Temp.	Rise with $Pb.Et_4$.
Benzene	690° C.	18° C.
Cyclohexane	535°	27°
Methylcyclohexane	470°	92°
Pentane	515°	75°
Isohexane	525°	46°
Heptane	430°	83°
Shell petrol	460°	82°
Ether	440°	65°
Alcohol	515°	9°
Acetaldehyde (iron vessel)	395°	110°
Acetaldehyde (silica vessel)	350°	170°
Amyl alcohol	490°	nil.
Amylene hydrate	640°	35°
Valeric aldehyde	320°	380°
Dimethylaniline	445°	nil.
Carbon bisulphide	120°	nil.

It would appear that the inhibitor acts on certain products of the primary oxidation of the hydrocarbons and tends to hasten that initial oxidation.

The process of combustion of the hydrocarbon begins by the formation of an unstable peroxide by the union of a molecule of the hydrocarbon and an active molecule of oxygen. This unstable peroxide may experience four possible changes, (*a*) it may radiate energy and form a stable peroxide; (*b*) it may break up into hydrocarbon and oxygen; (*c*) it may re-organise giving active products, e.g., $CH_3CH_3 + O_2 \rightarrow CH_3CH_2O_2 \rightarrow CH_3CHO + H_2O$; (*d*) collision may occur: if this is with a nitrogen molecule, the activity is diminished; but if with an active fuel molecule, further reaction will occur to form active products, thus establishing a centre of energy. Anti-knocks appear to act as inhibitors of the establishment of such centres.

From actual engine tests, it has been found that preliminary oxidation occurs during the compression stroke and that anti-knocks inhibit such oxidation. Engine knock or pinking appears to be due to inequality in the con-

ditions of the charge set up, particularly in regions of high pressure and temperature as in the neighbourhood of hot exhaust valves. W. N. H.

547. Flame Movement in Gaseous Explosive Mixtures. Part V.—The Propagation of Flame in Non-Spherical Vessels having their Three Dimensions of the Same Order of Magnitude. O. C. de C. Ellis. *Fuel*, 1928, VII., 336-344.

Further interesting series of photographs are published showing the spread of flame in pear-shaped and cylindrical vessels which bear out the general rule that the slackening in the rate at which the flame increases its volume reaches a critical point when the volume of the flame is equal to half that of the vessel. Ignition of an explosive mixture at two points in a cylindrical vessel whose diameter is equal to its length demonstrates the reluctance to merge always shown by flame surfaces which advance to each other through the same region of unburned gas, one flame sliding past the other. W. N. H.

548. Messel Memorial Lecture—Problem of Knocking and Anti-Detonants. C. Mouren and C. Dupaisse. *J. Soc. Chem. Ind.* 1928. 47. 33.

Anti-detonants are classed as anti-oxygenic agents. Several of these substances show anti-oxygenic action in low temperature oxidations—*e.g.*, iodine prevents the oxidation of benzaldehyde in the cold, as also do aniline, diphenylamine and lead tetra-ethyl.

The various theories of the action of anti-detonants are reviewed. The authors consider that their action takes place in the liquid phase, and that anti-detonants hinder the formation of peroxide by an anti-oxygenic action. The production of carbon during knocking is explained as the explosion of the peroxide, since the addition of one molecule of oxygen to one molecule of fuel is insufficient for complete combustion, and since high temperatures are developed in the liquid particles by the explosion of the peroxides the fuel is converted into a difficultly combustible mass of carbon. The increase in electrical conductivity of the gases of combustion during knocking is related to the ionisation which accompanies the autoxidation of phosphorus. A direct relation is found to exist between the tendency of a fuel to knock and the ease with which it forms cold flames; engine conditions which favour knocking (increase in compression, high oxygen content of the mixture, elevation of temperature and slow movement of the piston) favour autoxidation.

Experiments by the authors show that combustible liquids (especially those which possess detonant properties) auto oxidise in the liquid phase and give peroxides, and that anti-detonants show an anti-oxygenic action in the auto oxidation of heated auto oxidisable liquids. W. N. H.

549. Improvements Relating to Fuels for Internal Combustion Engines. C. J. Sims. E.P. 294,129. July 16, 1928.

Compression ignition engines suitable for aircraft must be of the high speed type and of low compression ratio. Ordinary fuels in such an engine show a considerable delay period in igniting; in order to minimise the delay period, various primers are added to the fuel. The following are mentioned as suitable primers:—trinitroresorcinol, nitrous and nitric esters—*e.g.*, methyl nitrate or amyl nitrite; azo compounds—*e.g.*, diazoaminomethane; haloid compounds—*e.g.*, iodoform; chlorates and organic perchlorates—*e.g.*, hydrazine diperchlorate; acetylenic compounds—*e.g.*, copper acetylide; superoxides—*e.g.*, benzene ozonide; nitroamines—*e.g.*, picramide; mercury

organic compounds—*e.g.*, mercury fulminate; also phenylnitramine and phenyl selenious acid. The quantity of primer required is small—*e.g.*, 5 per cent. in 95 per cent. of fuel. W. N. H.

Cracking.

550. Improvements Relating to the Pressure Cracking Treatment of Liquid Hydrocarbons. Beale, Coxon and Dunstan. E.P. 293,889, July 16, 1928.

The process is intended to prevent the deposition of carbon in pressure cracking apparatus by applying to the oil stream a high degree of turbulence whilst maintaining a feed of relatively low velocity.

The turbulence in the oil stream may be controlled independently of the rate of throughput during the time that the oil is under treatment.

This effect is accomplished by alternately surging the oil in opposite directions whilst passing through a cracking tube or a connected series of tubes by connecting a reciprocating pump to the inlet and outlet ends of the cracking tube or coil thus causing the liquid in the coil to move alternately in opposite directions to an extent corresponding to the capacity of the pump cylinder and at a velocity determined by the speed of the pump.

The process is particularly applicable to pressure cracking in the liquid phase and for single cycle treatment of residues, whereby a limited amount of light products and a non-viscous fuel are produced. The process may be carried out within a tube or coil without the use of a reaction vessel such as is usually employed for the completion of the treatment.

By the use of this process heat may be transferred rapidly and the liquid heated uniformly at a high pressure without danger, as the skin temperature is maintained at a minimum throughout the coil and the oil temperature is gradually reduced on active decomposition and rise of vapour pressure so that the maximum yield of light products may be obtained under a given working pressure without substantial vaporisation.

While still under pressure the oil may be rapidly cooled to arrest decomposition then flashed by reduction of pressure into a dephlegmator.

The time taken for the passage of the liquid may be only a few minutes, but the desired result is attained.

The conditions imposed do not favour the production of carbon, such as would be produced in known cracking apparatus, coking of the tubes is minimised and the small amount of carbon produced is to a great extent left suspended and is withdrawn with the liquid.

For these reasons continuous working of the process is to a great extent assured and the time necessary for cleaning is reduced.

In the experimental plant 1000 lbs. per sq. in. was maintained, the oil attained an alternating velocity of 12 feet per second, whilst the direct oil flow was only 1/30th to 1/20th feet per second. A Persian gas oil yielded 23 to 25 per cent. of Spirit with less than 0·01 per cent. of coke and a Persian residue yielded 15 per cent. spirit boiling below 200° C. with less than 0·1 per cent. coke. R. P.

551. Process and Apparatus for Treating Oil. D. G. Brandt. U.S.P. 1,681,321, Aug. 21, 1928.

The process consists of passing a superheated mixture of cracked gases and kerosine or gas oil through a body of oil contained in a vertical still fitted with baffles to distribute the gases and vapours.

The heat necessary for cracking is totally supplied by the hot vapours, so that the heating is internal, thus avoiding the bad effects of passing heat through a metal container.

It is claimed that by analogy to the laws of mass action the presence of the cracked gases tends to prevent the formation of more gas. Further, the gases act as an agitating medium and distribute the heat evenly through the oil to be cracked, besides permitting a lower pressure to be maintained in the cracking chamber.

The gases are circulated in a closed cycle, the excess being removed as necessary. R. P.

552. Apparatus for Converting Hydrocarbon Oils. O. C. Brewster. U.S.P. 1,681,658, Aug. 21, 1928.

From a vertical still vapours pass to a stripping column in which they are brought into intimate contact with fresh oil. From the stripping column the vapours pass to a condenser and receiving drum. Air is supplied at the bottom of the still and the heat required for the conversion of the oil is produced by internal combustion in this manner.

A heating coil is provided for bringing the contents of the still to a suitable temperature, about 350° C. at which active oxidation takes place and the supply of air is adjusted to maintain the temperature in the still at about 410° C. The oil is drawn from the bottom of the still by a pump and circulated through the heating coil then discharged into the upper part of the still.

A feed preheater in the form of a column receives the raw oil and raises it to conversion temperature, air being supplied to the bottom part of the preheater also. The preheated oil is supplied overhead to the stripping column, and passes through counter current to the vapours from the still. The fresh oil, together with the reflux oil, then flows downward into the still. R. P.

553. Treating Hydrocarbon Oils. H. W. Camp. U.S.P. 1,657,524, Jan. 31, 1928.

The patent describes a process and apparatus for distilling high boiling point hydrocarbons under pressure to obtain lower boiling point hydrocarbons. The apparatus provides an unheated still bottom portion into which the heavier residues from distillation tend to settle, and a scraping device adapted to remove adhering residues from the still walls. A residue draw-off, means adapted to clear the bottom portion of the still from settlings and an improved charging and run-back arrangement for the condensed still vapours are also provided. H. S. G.

554. Treating Hydrocarbons. C. S. Corning. U.S.P. 1,652,394, Dec. 13, 1927.

A method and apparatus are described for treating heavy oil continuously to secure lighter oils in which the lighter fuels in the form of vapours are treated to a cracking process with the application of heat to break up further the resulting products.

Crude oil is discharged into the first of a bank of retorts and circulates therein, passing out and on to a second and similarly a third bank of retorts. In the first bank it is initially heated and the more volatile gases are liberated and pass up into a header and thence into a chamber. Crude oil entering a retort is spread out into a thin stream and travels down and up a partition plate, thus exposing a considerable area to heated surfaces. Pyrometers are

provided for temperature measurement and by regulating the rate of flow of the oil an even temperature may be maintained. The gases evolved from the banks of retorts pass through a manifold into a chamber, where further heating takes place and cracking occurs. The vapours are drawn off under slight vacuum and condensed, distilled or otherwise treated.

H. S. G.

555. Process for Treating Oils. R. J. Dearborn. U.S.P. 1,658,077, Feb. 7, 1928.

This is a process of making gasoline which comprises passing a continuous stream of hydrocarbon oil through a heating zone, where its temperature is raised sufficiently to enable it to crack in contact with aluminium chloride in a separate reaction zone and without additional heat. The low boiling hydrocarbons formed are vapourised, the heavier components being condensed and returned to the reaction zone, whilst the remainder of the vapours are condensed to form gasoline. H. S. G.

556. Process for Cracking Petroleum Oil. C. P. Dubbs. U.S.P. 1,670,103, May 15, 1928.

The oil under treatment is heated in a coil and passes through a transfer line into a series of expansion vessels within which a differential pressure may be maintained.

Instead of passing the vapours serially through the expansion chambers they may be taken from the top of each to the dephlegmator. The chambers are provided with draw off lines and the heaviest part of the residue may be separated from the more suitable recycling stock.

The combined charging stock may be pumped into the dephlegmator, where it serves to cool the vapours and at the same time preheat the raw oil.

The oil may be subjected to a pressure of from 100 lbs. upwards and the oil so cracked as to convert from 50 to 75 per cent. into 225° C. end point gasoline. R. P.

557. Process of Treating Hydrocarbon Oils. C. P. Dubbs. U.S.P. 1,652,167, Dec. 13, 1927.

A process is described for automatically and continuously treating petroleum for the production of low boiling point products, and by means of rapid circulation of the liquid in the heating and distilling tubes permitting higher furnace temperatures to be maintained without injurious effect to the oil.

The material to be treated is drawn from a suitable source of supply by a centrifugal pump into preheating tubes and then into heating tubes preferably of 4 inches diameter. The heated oil passes upwardly into 10-inch distilling tubes, and when these have been filled to the desired level the pump is shut down and a valve on the line closed, remaining so during the rest of the run. Heat is applied to the furnaces and when the desired vapour pressure, which may vary from 50 to 500 lbs, has been created in the apparatus, a supply pump is started in operation. As distillation takes place, oil enters to maintain the oil level, any excess being by-passed back into the supply line, excess pressure being adjusted by means of an automatic relief valve.

A further pump is maintained in operation so that the oil contained in the heating and distilling tubes is continuously circulated in these tubes. The residuum formed in the distilling tubes is drawn off either intermittently or continuously. The vapours generated pass through lines to a manifold, any vapours condensed up to this point are drained back into the distilling tubes. The remaining vapours pass through condensing coils and into a

receiving tank. The oil temperature in the heating tubes may vary from 500 to 1000° F. as desired. H. S. G.

558. Process of Cracking Petroleum Oil. G. Egloff. U.S.P. 1,679,208, July 31, 1928.

The invention is intended to overcome or at least reduce the risk of carbon deposition and is accomplished by withdrawing the residue which accumulates in the expansion chamber and returning it along with a proportion of the uncondensable gases produced in the process into the vapour space of the expansion chamber in an atomised condition in order to obtain more effective heat transfer from the hot oil coming from the heating coil.

The uncondensable gas is mixed in such a proportion that atomisation of the residuum is effected. In this way practically all the oil can be recracked, producing a maximum amount of spirit leaving only coke to be removed from the expansion chamber and avoids the accumulation of quantities of residuum. R. P.

559. Process for Cracking Oil. G. Egloff and H. P. Benner. U.S.P. 1,659,398, Feb. 14, 1928.

A continuous process for cracking hydrocarbon oil consists in raising the oil to a cracking temperature while advancing in a restricted stream through a coil disposed in the heating zone of a furnace. The highly heated oil is delivered to one end of an enlarged reaction zone, wherein the vapours evolved from the oil initially flow at a relatively low velocity. The rate of conversion is accelerated by increasing the rate of flow from the inlet to the outlet of the reaction zone due to narrowing of the cross-sectional area. The vapours are led to a dephlegmator, where insufficiently cracked vapours are condensed and returned to the oil undergoing conversion. The uncondensed vapours are taken off for condensation and collection. During the process a self-generated superatmospheric vapour pressure is maintained on the oil. H. S. G.

560. Process for Catalytic Cracking of Heavy Hydrocarbons. E. Erlenbach. U S.P. 1,671,573, May 29, 1928.

A vertical still, which is heated by a furnace, is divided into two parts by a screen on which a deep layer of the catalyst is placed. The raw material is supplied at the upper end and a pump circulates the oil from the bottom half into the upper half of the still. Undistilled residue may be discharged from the bottom outlet.

The oil is maintained at a cracking temperature and the catalyst used is activated carbon. R. P.

561. Conversion of Hydrocarbons of High Boiling Point into Compounds of Lower Boiling Point. I. G. Farbenind. A.G. E.P. 270,314, April 28, 1927.

This is an improvement in the process described in E.P. 268,796. Production of low boiling point products from heavy hydrocarbon materials is carried out in the absence of any metals in the hot zone, but in the presence of such catalysts as do not give rise to the deposition of carbon, especially oxides of the heavy metals of groups three to six of the Periodic System. W. S. N.

562. Improvements in the Conversion of Hydrocarbons of High Boiling Point into Compounds of Lower Boiling Point. I. G. Farbenind. A.G. E.P. 268,796, 1928.

Heavy hydrocarbons are cracked by heating in absence of added water vapour or hydrogen, but in presence of metals which do not give rise to the deposition of carbon. The metals suitable for the process are cobalt, nickel, molybdenum, chromium, tungsten, manganese or their alloys, whilst oxides or sulphides may be used to augment the catalytic effect of the above materials. The catalyst should further be employed in a coherent condition and preferably the heat required for the reaction should be applied through the catalyst, e.g., electrically. The process is carried out under increased pressure to maintain the reacting liquid in the liquid phase. Five examples are quoted, the last being as follows: Crude mineral oil is heated in a chromium-lined still, fitted with an electric heater of chromium nickel alloy. The still is placed in a moderately heated bath, whilst the electric heater is raised to such a high temperature that active cracking is set up. The readily volatile products are distilled over and the asphaltic residue withdrawn periodically.

H. G. S.

563. Method of Treating Hydrocarbons. L. de Florez. U.S.P. 1,674,390, June 19, 1928.

The oil is passed through a preheater into a coil suspended vertically within a heating chamber. The tubes forming the coil gradually decrease in diameter as the oil flows from the cool end of the heating chamber to the hot end. The oil is vaporised in the relatively cool portion of the coil and cracking is effected in the hotter part. The resulting product is carried to an expansion chamber, where carbon and other residual products are deposited, thence to a condenser.

The initial feed pressure of about 125 lbs. is gradually reduced until the distillate receiving tank remains approximately at 15 lbs. The cracking temperature is about 610° C.

A portion of the gas produced in the process is utilised as fuel for heating and the remainder may be forced by a compressor into the oil stream, either before entering the heating coil, or in the heating coil, or after leaving the coil.

R. P.

564. Cracking Petroleum. L. J. Gary. U.S.P. 1,676,895, July 10, 1928.

Raw oil is supplied from storage by a pump to heating coils set in a furnace, passing thence via a transfer header into one of two expansion chambers, of which one is in use whilst the other is being cleaned. Each expansion chamber is provided with a residue draw-off pipe and vapour outlet, controlled by valves, vapours passing to a dephlegmator from which a reflux pipe leads back to the feed-pump. In connection with the dephlegmator is a condenser coil and receiver, provided with a valve, and leading to a vacuum pump. From each expansion chamber extends a short riser pipe, controlled by a valve, leading to a gas header controlled by a master valve. Condensable vapours passing into this header, condense and fall back into the expansion chamber, thus uniform pressure is maintained on the expansion chamber and heating elements, and in such case the dephlegmators and rest of the system may be maintained under a lower pressure. A second header controlled by a master valve and connected by branch pipes having valves to the dephlegmator operates in exactly the same way, maintaining a balanced pressure on the dephlegmators, expansion chambers and heating coils. The differential pressures thus obtained result in increased yields of light hydrocarbons.

H. S. G.

565. Process for Cracking Hydrocarbons. W. L. Gomory. U.S.P. 1,670,804, May 22, 1928.

This process consists of forcing a mixture of hydrogen and oil through a cracking coil maintained at a pressure sufficient to hold about 15 to 20 per cent. of the oil in the liquid state. The operation takes place at about 550° C. at a pressure of 150 lbs.

The mixture is pumped downwards through a heating coil then passes into a reaction chamber, where the body of oil is maintained in a quiescent state. The heavier part of the distillate is recycled with the raw stock.

R. P.

566. Treating Hydrocarbon Oils. W. S. Hadaway. U.S.P. 1,678,728, July 31, 1928.

If all the heat required for cracking be applied to cracking vessels it is difficult to prevent deposits of carbon and in this method the bulk of the heat is applied in a heating zone separate from the main cracking vessel.

The apparatus consists of a heating coil and a vertical conversion chamber. The oil from the heating coil enters a header at the bottom of the cracking chamber and passes up a number of tubes placed inside the wall of the chamber. These tubes are open at their upper ends and discharge the hot oil into the cracking chamber, which is provided with a central shaft carrying a number of propellers or agitators driven by a turbine situated within the chamber. The carbon formed is held in a state of suspension so that it may be easily drawn off with the liquid when desired.

R. P.

567. Apparatus for the Treatment of Hydrocarbons. E. R. Hamilton. U.S.P. 1,671,423, May 29, 1928.

The essential part of the apparatus consists of a number of concentrically arranged receptacles dividing the interior of a horizontal cylindrical still into a series of chambers. The latter are further divided by transverse partitions, which cause the oil to flow alternately up and down through a series of cells in each chamber. The still is heated externally and the oil is charged into the inner chamber. It flows through all the cells in that chamber, then passes into the next outer chamber and through all the cells that it contains, thence into the third chamber, and finally reaches the annular space between the still and inner receptacles or chambers.

R. P.

568. Oil Cracking Still. W. T. Hancock and J. W. Hancock. U.S.P. 1,680,541, Aug. 14, 1928.

A device which prevents heavy, tarry or carbonaceous matter from caking on the side of a still and maintains the heavy substances in suspension, allows them to be continuously withdrawn with the residue. In a furnace is located an inclined still tube with its vapour discharge outlet leading from its higher end and residue discharge pipe at its lower end. The tube carries a longitudinal central shaft by a bearing at one end and a stuffing box at the other, carried on the end of a pipe of reduced diameter projecting from the lower end of the still tube outside the furnace. Oil for treatment is supplied at a medial point and flows downwardly along the length of the tube, vapours and gases passing in the opposite direction. The longitudinal shaft has rigidly mounted on it a plurality of disks set at an acute angle. Balls are confined between these disks, whose peripheries are close to the inner surface of the still, and radial plates extending between adjacent disks carry the balls around in such a manner that they roll over the bottom part of the still wall and, at some point in their travel, drop against the still wall

with a hammering effect. At the same time the balls are moved back and forth longitudinally by action of the angularly set plates. The scrubbing and hammering effect of the balls keeps the solid matter in suspension in the residue. The construction is also such that, in their opposite longitudinal flow through the still, the residue and vapours are constantly brought to points close to the wall of the still, so that they are both entirely and uniformly heated to the predetermined temperature at which the still wall is maintained. H. S. G.

569. Controlling Destructive Distillation of Petroleum Oil. R. W. Hanna. U.S.P. 1,658,241, Feb. 7, 1928.

A process and apparatus for cracking petroleum are described in which the temperature and nature of the evolved vapours and entire reflux system are controlled by insulating the system from the varying effects of atmospheric conditions and dissipating heat to a medium independent of the cracking zone. By this means, the cracking zone may be operated at the optimum conditions desirable in cracking oil and more effective and higher rates of cracking can be maintained.

The apparatus comprises a still or evaporator, in which the body of oil undergoing cracking is maintained, provided with a cyclic heating system, consisting of a conduit with valves for regulating the rate of flow, communicating with the evaporator at one end and leading through a pump to a heating coil set in a furnace and thence through a conduit back to and re-entering the still at the opposite end. A vapour line leads from the evaporator to the bottom of a tower provided with baffle plates. An oil supply line leads through preheaters and discharges into the centre of the tower. A vapour line leads from the top of the tower to near the bottom of the heat dissipation member, which consists of a closed cylinder with tube sheets or partitions through which is circulated oil for preheating or cooling water. The vapour line leads out to a condenser and storage passing through a seal to trap any condensate from the dissipation member, which is returned to the top of the tower and discharged on to the uprising vapours.

By bringing the feed stock and reflux from the heat dissipation member into the vapour tower there is provided a three phase vapour separation: (*a*) The rough scrubbing in the tower below the point of feed, (*b*) the scrubbing by the condensate from the heat dissipation member, and (*c*) controlled cooling effect of the heat dissipation member, and a more thorough sifting of the higher boiling fractions attained. By this means the destructive distillation of petroleum may be carried on under the most efficient conditions, the controllable feature of heat dissipation member making the change in rate of firing, rate of feed or change in other variables unnecessary for the production of desired distillate. H. S. G.

570. Art of Cracking Hydrocarbon Oils. E. C. Herthel. U.S.P. 1,677,773/4/5/6, July 17, 1928.

The oil to be cracked is subjected to distillation under pressure at cracking temperature, the vapours from the still are subjected to a refluxing operation, the reflux oil is returned to the still, and the remaining vapours are condensed. Fresh oil is supplied continuously to the still and unvaporized oil containing pitch is withdrawn. The rate of feed and run off are regulated so that the concentration of pitch is kept below the saturation point. The pitch-laden oil is expanded by reduction of pressure and is separated into an oil fraction and a heavy residue. The oil vapours separated from the pitch are passed into a tower to which fresh oil is introduced. The fresh oil is heated and the

vapours are cooled, and uncondensed vapours corresponding to the vapours from the reflux tower of the still are passed through a condenser to unite with the light product from the still.

The heavier components of the vapours along with the unvaporized portion of the fresh oil are then introduced into direct contact with the vapours from the still.

A part of the condensed pressure distillate may be returned to the still dephlegmator at a point above the introduction of the mixture from the second tower to maintain the temperature of the escaping vapours at the desired temperature and control the refluxing operation. Also, part of the mixture may be introduced into the still directly at practically the same temperature at which it is withdrawn from the second tower.

The operation of the second tower may also be further controlled by introducing pressure distillate above the point at which fresh oil is introduced, in such an amount that the escaping vapours are of the desired quality.

R. P.

571. Conversion of Hydrocarbon Oils. E. T. Hessle and W. Lelgemann. U.S.P. 1,658,985, Feb. 14, 1928.

The apparatus consists of a cast-iron reaction kettle formed with a partition, perforated and serrated at its lower end, dividing the interior of the kettle into two compartments, one approximately one-eighth the volume of the other. On the wall of the kettle and on one side of the partition are a series of ribs supporting a series of movable plates. A removable cover is provided, normally held in place by bolts, and provided with temperature and pressure measuring devices and an outlet conduit. Mounted on the wall of the vessel is a fog producing device in which a homogeneous fog is produced by impact between oil and a gas. A needle valve is provided for maintaining the passage clear of obstruction. Look glasses are provided in the wall of the vessel.

In operation, the kettle is heated to a temperature between 350-450° C., sufficient to melt a catalyzer composed of a tin-antimony alloy, and oil and gas supplied to the fog producing device in order to produce a homogenous fog controlled by viewing through the look glasses. The fog passes through the molten catalyzer by way of the serrations and perforations at the end of the partition, escaping into the space above from which the vapour is led to a suitable condenser.

H. S. G.

572. Refining Mineral Oils. E. T. Hessle. U.S.P. 1,661,826, March 6, 1928.

This is a process for the catalytic decomposition of hydrocarbon oils by passing a fog of the oils through a molten catalyst containing tin, or an alloy of tin and antimony, maintained at a temperature below the normal boiling point of the oils. It is intended further to subject the oils in the presence of hydrogen to successive decomposition and hydrogenation catalysts, whereby saturated hydrocarbons of lower boiling points are produced with minimum carbon and fixed gas formation. The hydrocarbon oils to be treated are pumped into a steam jacketted tank, and thence through a pipe heated by super-heated steam into the first of a series of vessels set in a long and narrow shaped furnace. In the first vessel, the atomizer, the oil is directed against a counter current of hydrogen and superheated steam under three atmospheres pressure and thus becomes finely atomized. The fog is conducted by a pipe into the bottom of a reaction vessel partly filled with molten catalyst, preferably tin or tin-antimony alloy. Baffles disperse the oil fog so that it passes slowly upward through the catalyst which is maintained in general

between 350° and 450° C. The vapour released passes on into a further similar reaction vessel, where conversion is completed, and from which the vapours pass on to a vessel partly filled with steel turnings which act as a catalyst to hydrogenate any unsaturated compounds present. From this vessel the vapours are taken to a column separator, the condensate from which is adapted for use as light lubricating oils. From the separator, the uncondensed vapours pass into a vessel filled with copper and nickel cuttings, where further hydrogenation takes place, then on into a tank filled with material consisting of 90 per cent. fuller's earth and 10 per cent. nickel, finely dispersed throughout. The vapour from this tank is led into a further tank, where it is forced through lignite coal, having finely dispersed metallic nickel and 2 per cent. aluminium oxide formed thereon. The vapours pass to a fractional condenser, where products having a boiling point between 150° and 250° C. are condensed, and the uncondensed vapours pass to a coil where gasoline is removed. Any uncondensed vapours from here pass to a coil immersed in brine at minus 10° C., and any vapours from here pass on into a tank filled with granulated coal. Dampers provided in the furnace setting enable each of the reaction vessels to be maintained at predetermined temperatures. A 70 per cent. gasoline yield from an unrefined mineral oil having a boiling point above 350° C., which can be increased to over 90 per cent. by returning the heavier condensates, is claimed. H. S. G.

573. CATALYTIC APPARATUS. E. T. Hessle and W. Lelgemann. U.S.P. 1,661,827.

An improvement on the apparatus described in U.S.P. 1,658,985 is described for the catalytic conversion of petroleum oils and residues into lighter hydrocarbons. Means are provided for removing residues of oil and carbon formed or deposited on the surface of the catalyst. An equalizing vessel is also provided to maintain the body of the catalyst at the desired height in the reaction vessel.

The apparatus consists of a cast-iron reaction vessel provided with a longitudinal partition extending from the top to near the bottom of the vessel and dividing it into two unequal compartments. A removable cover is provided with pressure gauge and thermometer well. An oil fog producing device extends through the wall of the reaction vessel into the left hand or smaller compartment and is adapted to be connected to oil and gas supply pipes, which communicate with opposed nozzles, the fog being produced by the opposing streams of oil and gas issuing under pressure. This fog of oil is produced under sufficient pressure to cause it to pass down through a body of molten catalyst, preferably tin or tin-alloy, with which the reaction vessel is partly filled, and up into the compartment on the other side of the partition. The decomposition products discharge through a pipe in the cover, which has a curved baffle spaced to prevent the catalyst from being carried out. Perforated hozizontal baffles in the larger compartment serve intimately to mix the fog and catalyst. An equalizing vessel, comprising an upright cylinder flanged at each end, is connected to the reaction vessel by means of a bent pipe from the bottom of the equalizer to the bottom of the reaction vessel. A side communicating pipe is also provided at a height to permit the desired height of catalyst and permit gravitational circulation. As the oil fog passes through the molten catalyst, it depresses the catalyst in the left hand compartment of the reaction vessel, causing it to rise in the right to the level of the communicating tube. Thus the height of the catalyst is kept regulated. During the progress of conversion of oil into lighter hydrocarbons, oil residues and carbon form on the surface of the catalyst and

pass over into the equalizer from which they can be skimmed off through a draw-off line. A floating indicator is provided to ascertain the depth of the oil layer. H. S. G.

574. Improvements Relating to Protecting the Interior of an Oil Cracking Retort. A. C. Holzapfel. E.P. 291,585, June 7, 1928.

The following substances: commercial waterglass 45 parts, water 28 parts, magnesite 5 parts, slaked lime 2 parts, fireclay 10 parts, are mixed and form a paint which is claimed to prevent corrosion of cracking plant. It should be applied to the metal surfaces of cracking stills as soon as possible after preparation as it tends to thicken. Two or more coats should be applied preferably one coat every two or three weeks.

The composition is adhesive to steel and not only withstands a temperature of around 550° C., but is indurated and improved by this treatment. R. P.

575. Pyrogenesis of Petroleum Products. F. A. Howard. U.S.P. 1,676,694, July 10, 1928.

A mixture of hydrocarbons is delivered under substantial pressure, e.g., 450 lbs., and at a temperature of 860° F. into the base of a primary drum, where a temperature of 770° F. is maintained. The mixture of products discharge from an overflow pipe at the top of the drum into the base of a secondary drum, emerging when this is full through an outlet line leading to an expander, separator or cooler. By means of the introduction of a stream of cool hydrocarbon oils under pressure into the base of the secondary drum, the temperature is maintained well below that in the primary drum. A reverse-circulation pump withdraws oil from the base of the secondary drum and discharges into the base of the primary drum, and by control of this circulation, any desired temperature is maintained. By this means efficient control is maintained leading to decreased production of gaseous products. H. S. G.

576. Treating Oils. F. A. Howard. U.S.P. 1,676,826, July 10, 1928.

A process of treating hydrocarbon oils is adapted to stocks not hitherto readily subjected to heat conversion treatments owing to their tendency to form heavy coke deposits. Cold fresh stock is fed from a reservoir, through a pump, meter, first and second heat exchangers, and into a chamber where its natural naphtha is removed. The residue is pumped through a pipe to a fired coil, emerging at a temperature about 800° F. at a velocity sufficiently high to prevent coke deposition. Steam may be injected into the oil stream to maintain the desired velocity. From the coil the oil enters a drum mounted in a setting so as to receive the flue gases from the firing of the coil. Here the velocity is considerably reduced and coke is deposited. The oil leaves the drum at about 720° F. by a pipe at its highest point, passing through a valve and through a coil maintained in a still. A considerable pressure is maintained within this system, so that when the oil passes through a pressure control valve into a container, the large drop in pressure results in considerable vapour evolution. The vapours pass out through the heat exchanger to a condenser. H. S. G.

577. Cracking Hydrocarbons. F. A. Howard. U.S.P. 1,681,899, August 21, 1928.

An improvement on specification 1,415,232 of Ellis. The apparatus comprises a furnace in which is located a heating coil positioned to absorb radiant heat. Oil to be treated, preferably topped crude, passes through this coil and discharges into a still, having been heated sufficiently to allow

substantial vaporization of desired constituents. Further heat is supplied by normal firing, and distillation is carried as far as possible whilst allowing the residue to be drawn off continuously or intermittently as desired. A pressure in this still of between 5 and 150 lbs. per sq. in. is desirable. Vapours proceed to a second coil in the furnace positioned so that the first coil protects this one against the radiant heat. The vapours are heated up to about 825-950° F. and discharge into an enlarged passage-way or drum, where a pressure of from 15-150 lbs. per sq. in. is maintained. From the drum vapours proceed to a rectifier and thence to a condenser and receiving drum. To facilitate rectification, condensate is drawn off from the receiver and pumped into the upper part of the rectifier and passes down countercurrent to the vapours. The heavy reflux from the bottom of the rectifier is returned through a suitable trap to the still for recycling. H. S. G.

578. Oil Still. E. W. Isom and A. C. Vobach. U.S.P. 1,676,204, July 3, 1928.

An improvement in connection with oil cracking stills of the general character described in U.S.P. 1,285,200. According to the present invention the heating tubes are provided with centrally located cores extending entirely through the heated portions and consisting of twisted rods. The extended helical configuration of the surface materially increases the velocity of the circulating oil, the centrifugal action assisting in preventing the accumulation of carbonaceous material on the heated tube walls and assuring an increased rate of heat transfer. H. S. G.

579. Cracking Hydrocarbons. E. W. Isom and C. L. Parmelee. U.S.P. 1,675,558, July 3, 1928.

In cracking operations, in order to avoid carbon formation, super-saturation of the oil with heavy tarry matter is prevented by adding to the oil being cracked in the heating zone a solvent oil, such as gas oil or kerosine. A preferred apparatus embodying the invention consists of a firebox, stack and zig-zag flue divided into a number of heating chambers, each containing a group of coils extending horizontally across the furnace. Material to be treated is pumped to the last coil of the series, where it is preheated by the relatively cool gases before passing to the other coils, where cracking of the oil is substantially completed. It then passes to an expansion chamber through a pressure reducing valve. The cracking action in the tubes of the first pass releases a certain amount of heavy tarry soluble matter, and before entering the second pass of the furnace, where the additional heat would result in saturation of the oil with heavy tar and carbon formation, a stream of solvent oil is introduced of such character and amount to maintain such material in solution. An additional stream of solvent is introduced before each successive pass of the furnace, thus preventing carbon formation in these tubes. Preferably the solvent oils consist of fractions separated from the oil flowing from the cracking device.

In the expansion chamber, the lighter components of the synthetic crude are volatilised by the heat contained therein. Tar flows through a pipe to a cooler and tar holder. Vapour from the expansion chamber passes to a condenser through heat exchangers employed for preheating the solvent supplied to the cracking coils. From the condenser, the condensate may run to storage or to the re-run still, where four cuts are made, the first going to the gasoline storage tank. The third cut, of gravity about 38° B., is used as the solvent introduced between the first and second cracking coils, to which it is pumped via the heat exchanger. The second cut, of gravity

about 41° B., may similarly be used as the second injection of solvent at a point between the second and third cracking coils. The remaining cut may approximate gas oil in composition, and is conveyed to the storage tank from which the stock to be treated in the cracker is taken. H. S. G.

580. Process for the Distillation and Cracking of Hydrocarbons. H. J. Jansen. U.S.P. 1,672,459, June 5, 1928.

The process consists of passing the hydrocarbons through suitable metals or salts or a mixture of both in a molten condition. The best results in many respects are obtained with a lead bath covered by a layer of zinc chloride.

The process can be applied directly to a distillate, but if heavy asphaltic oils are used the process may be modified to treat the oil in two steps. In the preliminary step the heavy oil is passed through a molten mass, having only a small free surface so that the oil is decomposed into a heavy residue, which takes up the carbon and is quickly discharged from the plant, and a vaporized part which is further cracked by passing it through a second molten mass heated to cracking temperature.

The apparatus for both stages consists of a horizontal container entirely filled with the molten mixture, containing a dome at one end having a vapour outlet, and an oil inlet at the other end. R. P.

581. Process of Treating Oils. H. C. Kirk. U.S.P. 1,676,230, July 3, 1928.

An externally heated still is filled with oil to a convenient level and brought to the desired temperature. Further oil is now pumped into the still through three inlets: (1) leads to an injector with downwardly directed nozzles submerged just below the upper surface of the oil in the still; (2) leads to a spray ring placed near the lower level of the oil with jets arranged so that oil is forced upwardly; (3) connects to a pipe arranged vertically and paralleling the conical bottom of the still provided with openings adapted to project oil into the body of oil below the spray ring and give such oil a circulatory movement. By such means a uniform rate of circulation of oil is maintained at all times and no portion is allowed to come to rest. This eliminates any formation of hard, adherent, coke-like deposits. Any carbon formed is soft and mud-like and is kept in constant circulation until withdrawn through the tar line. A further point is the elimination of all stirrers or other mechanical devices. Vapours and gases are drawn off through a pipe to a dephlegmator, condenser and storage vessel in the usual way, and the apparatus is adapted for use under high cracking temperatures and pressures. H. S. G.

582. Cracking Oils. C. L. Parmelee and E. W. Isom. U.S.P. 1,675,575, July 3, 1928.

This is a description of an improved method of operation of tubular cracking stills by the introduction into the coils of the tubular still a solvent oil, which mingles with the partially cracked oil and permits a higher degree of cracking to be continued over a longer period, with reduction of the danger of carbon depositing upon the tube walls.

A preferred apparatus is as follows:—A series of three coils is arranged in a single furnace together with a preheating coil. Feed oil is supplied to the preheater coil and flows thence to the first cracking coil and on to the second and third. Provision is made for the introduction of a solvent oil into the cracking still at points between the first and second, and second and third coils. Cracked oil leaving the tubular still passes through an expansion

valve to the expansion chamber, where the heat of the cracked oil is used in vaporizing the lighter constituents which pass off through heat exchanges to a condenser, gas separator and storage. The unvaporized portion of the cracked distillate is drawn off to the cooler and pumped to storage. The solvent oil may be preheated to cracking temperature or below before introduction. Pressure in the apparatus can be regulated by the valve between the still and the expansion chamber, and will be influenced by the rate of feed of changing stock or solvent oil entering the still. H. S. G.

583. Improvements in Cracking Hydrocarbon Oil. R. T. Pollock. E.P. 287,344. 1928.

Oil is cracked under suitable conditions, the vapours subjected to dephlegmation and the reflux distilled by supplying heat other than that obtained from the heated oil, to remove light fractions. The distilled reflux is then returned to the cracking system. H. G. S.

584. Apparatus for Cracking Hydrocarbon Oils. R. T. Pollock. U.S.P. 1,670,122. May 15, 1928.

The apparatus consists of an elongated furnace with a baffle wall above, and a number of tubes are arranged in a super-imposed flue. These tubes are connected to headers at each end, the oil being pumped into one header and removed from the other into a vapour chamber situated above the tubes and separated from them by a second baffle wall. The flue gases travel from one end of the furnace along the tubes then pass over the vapour chamber. R. P.

585. Process for Cracking Petroleum Oil. R. T. Pollock. U.S.P. 1,658,415. February 7, 1928.

This is a process for separately cracking reflux condensate produced in cracking petroleum oil. Petroleum fuel oil, or other oil, is fed continuously to heating tubes set in a furnace where its temperature is raised to 750° F., and while substantially in liquid phase, is passed to the vapour chamber and thence to the first reflux condenser. Uncondensed portions pass thence to a partial condenser and still uncondensed portions pass to a further condenser and receiver. A pressure of 150 lbs. is maintained in the above systems. The first condensate is pumped to a second heating coil and there subjected to a temperature of 800° F. passing into a vapour chamber and dephlegmator. This part of the system is kept under 200 lbs. pressure. Condensate from the dephlegmator passes into a third similar heating and condensing system, where a temperature of 850° F. and pressure of 250 lbs. is maintained. From the foregoing it will be seen that the raw oil and different cuts of reflux condensate are all treated under different pressures and temperatures. H. S. G.

586. Cracking Oil. R. T. Pollock. U.S.P. 1,676,924. August 10, 1928.

Heated oil, raised to cracking temperature, rises vertically through a line in the centre of a vaporizing chamber shell. Near the top of the chamber, this line reverses its direction and travels downwardly in a spiral coil, passing off as a draw-off line near the bottom of the chamber. Tapped into the spiral at intervals are a number of vapour risers extending upward above the top of the reversal. Oil entering the vertical pipe rises and flows downwardly through the spiral, and since it is at cracking temperature, vapours are released from the oil body and pass out through the vapour risers to a condenser. The unvaporized fractions, together with carbon and heavy residual material, pass out through the bottom of the spiral tube, any accumulation of carbon

or deposit being prevented by the continuous flow. Vapours escape freely, and in addition the circulation of heated oil maintains the vaporizing chamber at a high temperature, facilitating redistillation of any oil fractions which may tend to reflux. H. S. G.

587. Cracking Oils. G. L. Prichard, Assr. to Gulf Refining Co., Texas. U.S.P. 1,676,207. August 3, 1928.

A method is given of cracking oils, producing more uniform results and less carbon deposition. This is obtained by providing stirring mechanism in the still by which means coke tends to form in granules and not adhere to the heating elements. The apparatus consists of a still provided with external or bottom firing. At its top is a vapour outlet leading to an air-cooled chamber from which a vapour line passes to another air-cooled chamber beyond which is an outlet provided with a loaded valve. Vapours are led through a conduit to suitable condensing apparatus beyond which is a pressure valve leading to a release chamber. The air-cooled chambers are provided with an intercommunicating run-back leading back into the still.

The bottom of the still is dish-shaped. Across the still at a distance from the bottom is an angle-iron bracket supporting a thrust collar through which passes a vertical shaft to which is connected chains for dragging across the bottom of the still. A plate on top of the still forms the support for the cooled bearing, consisting of a casing, provided with inlet and outlet for cooling fluid, through which the shaft, surrounded by metallic packing, passes. At the bottom of the still is a draw-off for tar and granular coke. Motion is given the stirring mechanism by means of gears. The still is charged to any height with oil to be cracked, the stirrer kept in steady motion and heating started. The pressure is kept at the desired point by adjustment of the rate of heating and the pressure valve. During the heating operation oil is kept flowing into the bearing casing, maintaining it cool and keeping a tight joint round the stirrer. If desirable the oil may be under a pressure equivalent to, or greater than, that in the still, by which means leakage is impossible. H. S. G.

588. Cracking Mineral Oil. J. M. Schoonmaker. U.S.P. 1,678,126. August 24, 1928.

Every petroleum oil or fraction has a definite critical temperature for a given pressure, below which cracking does not take place to any appreciable extent, and above which cracking proceeds rapidly. By increase of pressure this temperature may be raised from 10° F. to 50° F. to a maximum critical temperature beyond which increase in pressure will no longer prevent cracking. If an oil is heated to a point between its cracking point under normal pressures and its maximum critical cracking point, and is then suddenly allowed to expand, cracking occurs with almost explosive rapidity. This reaction is promoted by the application of heat at the moment of expansion.

A suitable apparatus for carrying out the above reaction consists of a tubular heater set in a brick furnace and through which oil is pumped at a pressure of abour 1,500 lbs. per square inch. The oil at a temperature of from 700° F. to 900° F. passes to the pyrolysis tube heated by a gas or oil burner where the oil attains a temperature of about 1,050° F. and discharges into a separating chamber. This resembles an ordinary dephlegmating tower and contains baffles for the separation of fixed gases and vapours from solid and liquid residues. It is provided at its lower end with a ring burner in order to provide the heat which would otherwise be lost by the expansion to prevent condensation and to keep the residues liquid. 820° F. to 840° F. is a suitable temperature for this chamber. Tarry accumulations are drawn off from the

bottom of the tower. By means of a high-pressure pump vapours are withdrawn from the top of the tower and delivered to the condenser under a pressure of from 100 to 200 lbs. per square inch.

Two cracking tubes and two towers may be fed from a single heater so that the process may be made continuous, one set being cleaned whilst the other is in use. Actual carbon deposition is very small, since the nature of the process produces carbon in such a fine form that the greater part is absorbed by and discharged with the residues. H. S. G.

589. Improvements Relating to the Conversion of Heavy Hydrocarbons into Hydrocarbons of Lower Molecular Weight. A. D. Smith and J. Perl. E.P. 289,673. May 3, 1928.

The invention relates to the production of light hydrocarbons from heavy hydrocarbons by cracking in two or more stages with intermediate cooling and with intermediate separation of desired products. In the process the deposition of carbon is prevented by the continuous removal of the highly-polymerised compounds from the reaction zone before their decomposition into free carbon, so as to leave a residue which can be further cracked without separation of carbon. Further, by continuously cooling a portion of the reacting medium before separation of the polymerised hydrocarbons takes place, precipitation of the soluble polymerised compounds is effected before saturation occurs. This is obtained by an interchange of heat between the freshly-charged oil and the saturated partially converted oil remaining after vaporization of the light hydrocarbons.

Another object of the invention is to increase the total yield of light hydrocarbons by subjecting the stock and the recycle oil separately to temperatures and pressures best suited to obtain the desired result. To prevent carbon formation in the latter stage of cracking, means are provided for controlling the contact interval between the liquid and the reaction plates. Polymerisation of sulphur compounds is stated to occur and these may be precipitated along with the heavy polymers, thus preventing decomposition and combination with the metal of the cracking plant.

Prior to a description of the plant the theory of cracking reactions is discussed.

The plant consists of two sets of heating coils. The first coil is supplied with crude stock and the partially cracked oil passed into a vertical still where dephlegmation of the gaseous products is effected and further cracking developed by means of hemispherical conversion plates, which are perforated and mounted on horizontal parallel shafts which may be turned to close the cross-section of the still. At the base of the vertical still a large outlet pipe allows the liquid descending to be cooled by the incoming crude oil. Thence the cooled liquid descends to a precipitation chamber and the same solid asphaltic tars are drawn off through a waste line. The de-saturated, cooled and partially converted liquid to be recirculated returns to the second heating coil, whence it is united with the stream from the first coil and discharged into the vertical still thus, completing the cycle of operations.

The vertical still is provided with a vapour outlet which connects to a condenser.

In the primary coil a pressure of about 300 lbs. is maintained and a temperature of about 450° C., depending on the cracking stock. The secondary coil and the still may be at 375° C., but the maximum yield of light products will be proportional to the extent of cracking, this being highest with the longest contact of the reacting phases during passage over the hemispherical plates in the still. R. P.

590. IMPROVEMENTS IN OR RELATING TO PROCESSES FOR CRACKING VEGETABLE AND MINERAL OILS. Soc. Anon. le Carbone. E.P. 290,060. May 10, 1928.

The process consists in cracking oils by bringing the vapours of the raw stock into contact with activated carbon in the shape of compact blocks of an essentially porous nature. These blocks act simultaneously as filters or atomisers and avoid losses of the material such as occur when the material is in powder or granular form. R. P.

591. PLANT FOR THE MANUFACTURE OF LIQUID FUELS. Soc. Int. des Procédés Prudhomme Houdry. E.P. 267,512. May 10, 1928.

The present invention is an improvement on previous patents Nos. 238,931, 238,805, 267,138, and comprises means for the introduction of fresh hydrogenating gases before entry into the intial catalysers in order to maintain a suitable reducing atmosphere and for the production between catalysers of nascent hydrogen.

It also provides means for the return of the gases to the apparatus.

This plant is designed for the manufacture of liquid fuel and comprises a gas-generating apparatus for the pyrogenation, gasification or distillation of carbonaceous materials, a purifying and desulphurizing apparatus, and a series of catalysers wherein hydrogenation of phenols and carbon monoxide takes place. A breaking up of the hydrogenated products with the production of nascent hydrogen follows, then polymerisation is effected. R. P.

592. APPARATUS FOR TREATING OILS. F. E. Wellman. U.S.P. 1,672,668. June 5, 1928.

A circular series of cracking tubes are mounted within a circular furnace close to the inside wall, and project through the top of the furnace. Four vaporizing drums are provided around the top of the furnace, each of which is connected with several of the cracking tubes. A common vapour line is connected to the drums for the removal of the products and outside circulating pipes connect the drums below the liquid level.

The cracking tubes are connected to a manifold extending round the outside of the furnace to supply the raw oil.

Heat is applied to the cracking tubes by a rotating burner, and a set of baffles within the furnace distribute the heat among the cracking tubes. R. P.

See also Abstracts Nos. 489, 500, 504, 512, 513, 514 and 516.

Oil-Field Technology.

593. HYDRAULIC LONG STROKE DEEP WELL PUMPS. D. Moore. *Oil Field Engineering*, 1928, No. 6, p. 52.

The deep well pump described in this article is installed in the Seminole field at a depth of 3,400 ft. The equipment consists of a steel cylinder, 15 ft. long, 3¾ in. inside diameter, 5¼ in. outside diameter, fitted to a steel trellis, and inside of it is operated a piston rod 18 ft. long. The trellis is 56 ft. long and stands 26 ft. high, being supported by two A frames of 2 in. pipe.

A 45 h.p., 900 r.p.m. electric motor, mounted on a concrete foundation, is directly connected to an oilgear hydraulic pump through a universal joint. Either gas or steam power could be used instead of the motor.

To operate the pistons, oil is forced at the rate of 15,000 cubic inches per minute, through 2 in. lines into each end of the cylinder alternately, by the pump at 1,500 lbs. pressure, which causes the raising of the rods and lowering

of the counterweight attached to the opposite end of the piston rod. Each stroke is automatically reversed. The counterweight, attached by means of ⅝ in. wire rope running over 42 in. pulleys, weighs 3 tons.

The stroke can be varied, by moving the control mechanism, up to 25 ft. in length. The pump described operates with a 12 ft. stroke at an average speed of eight stokes per minute and delivers about 600 barrels daily through 2½ in. tubing. T. W.

594. Development of American Equipment Applicable to Foreign Fields. R. R. Robinson. *Oil Field Engineering*, 1928, No. 6, p. 23.

The author in this article only deals with the major equipment used in drilling and completing wells without minimizing the importance of smaller tools and accessories. The U.S.A. manufacturer is particularly favourably situated in dealing with suggestions for improving and advancing the development of equipment, from home and foreign countries, inasmuch as the trying out of new ideas takes place on his doorstep. Many of the improved machines have been built to the order of foreign operators, whose needs have been an important controlling factor.

Steel-made derricks were just coming into use a few years ago, and are now found in practically every country where drilling is carried on. A new type is that in which the upper portion is so designed that it may be lowered intact inside the heavy base and used as a pumping derrick, while the bottom portion may be moved to another drilling location.

To-day the high-torque, multi-speed, gas protected electric motor stands up under service far beyond the capacities of the early electrical applications, with reserve power greatly exceeding that of the steam engine and without the sacrifice of flexible control. The electric motor has also made possible many improvements in driving mechanism, notably on all geared, six-speed, motor-driven unitary drawbacks. Gear reductions being necessary for use with electric motors the oil industry has awakened to the economies of the high-speed prime mover, and the trouble-free operation of the various gear reduction units has emphasized the time lost from the use of belted and chain-driven units. Consequently various adaptations of the high-speed internal combustion motor have been introduced. The development of efficient and dependable clutches has been another inducement to the more general use of gas engines.

Slow-speed engines, both gas and steam, have also undergone extensive improvement; most important are the complete enclosure of all working parts and automatic lubrication. Another development is the application of the full Diesel engine of three, four and six cylinder units, to both cable and rotary drilling. These units can be operated on crude oil.

Among the slush-pump improvements is a noticeable tendency towards lengthening the stroke, the latest being 20 in. Pressures for water ends are now designed for 1,500 to 2,000 lbs. per square inch, and in the later designs a feature is the lowering of the suction valve. Some water ends are split and interchangeable side for side to allow for more economical replacement. Owing to increased water end pressures steam cylinders are now built for steam pressures of 200 and 250 lbs.

In the six-speed, all geared unitary type of drawworks the drum and line shaft are mounted on roller bearings. Herring-bone gears are used for the final reduction on the low-speed, and planetary spur gears on the high-speed side. Another drawworks unit has an extra large diameter and length of drum, and to provide for the necessary speed changes an auxiliary line shaft is placed behind the drum close to the floor. This unit, of especially heavy construction, was designed to decrease the winding and crushing

of the wire line, together with more efficient operation. On some types forged brake drums are furnished to provide safety against the stresses due to high speed and heating.

The double-deck type with casing sheaves on the upper deck has almost superseded the old type of crown and travelling blocks; 36 in. and 48 in. casing sheaves are used, the block being raised higher with the new design owing to alignment of sheaves. When drilling is completed the upper deck may be removed, and the lower deck left in place for rod and tubing hoisting, or a special pumping crown block of lighter construction substituted. Owing to derrick space limitations travelling block sheaves have not gone beyond 36 in. diameter.

Few radical departures have been made in the usual construction of lines. A few types have been manufactured from specially shaped wires designed to give greater drum and sheave riding surface. The one outstanding development is the so-called preformed rope in which the wires are formed to the shape they will occupy in the finished rope. These lines are exceptionally free from kinking and spreading. Such lines are easy to socket and handle, and special fittings are applied giving a connection for which unusual strength and service is claimed.

Pinion drive shafts on the new rotary table are mounted on roller bearings. Cast iron parts in rotary tables are almost entirely eliminated, except in cases for light service, and electric and forged steel parts substituted. The double table rotary or pipe screwing type, as it is called, makes it possible to make up the casing as well as break it out, being quite a time-saver in running casing, as well as reducing labour in running in the drill pipe.

To prevent the great amount of wear, when drilling through hard formations, between the drill pipe collars and tool joints and the casing, many drill pipe strings are now fitted with ball-bearing tool joints, and recently a rubber casing protector ring of tough resilient rubber has been introduced. This is slipped over the tool joints and grips the pipe, and being soft does not wear the casing, while preventing contact of the collar or tool joint against the pipe, and is easily changed when worn. Another device is the casing turner which is installed upon a string of casing above the cementing zones and makes it possible to rotate the upper section of the pipe so as to present a new surface to the wear points and prevent long-continued wear in any particular portion of the casing surface.

Rotary bits have been given an impetus in the last few months by the growing use and excellent results obtained from the use of hard metal inserts. These inserts are adaptable to most all present type of bits. Some have interchangeable and renewable cutters or cutting heads which may be discarded when worn. Core bits have been greatly improved through the application of the hard metal inserts. Cleaner cutting and more uniform cores has been the result. One core barrel is a recent design in which the inner barrel is stationary. Another inner barrel is split for easy removal of the core.

Core barrels have also been developed for cable tools.

The uncertainty of results with the early rotary under-reamers, and the ever-present possibility of losing them in the hole, kept them from general use. Present designs have been developed in which the cutters are positively expanded and in which collapsing when desired is practically assured. It is also the practice to run a straight reamer behind the bit. The side support thus given the drill pipe helps to produce a straighter hole.

Few of the modern drilling rigs are without a weight indicating device to indicate and record the weight on the hook. Favourable devices are those adapted to be attached to the dead line and thus indicate the line load.

Such instruments are of immense value in regulating the weight on the bit and in guarding against excessive overloading of hoisting equipment or the derrick.

The rotary jar is being used frequently in the drilling string, especially behind core barrels and under-reamers. This jar is designed so as to transmit all the necessary thrust and torsional loads. If sticking should occur the tools may be jarred loose.

Another device in long strings of drill pipe is the float valve, a check valve placed in the drill pipe to reduce the weight on the hoisting apparatus by "floating" the string. These valves are designed to close against pressure from beneath, but to open for the circulating fluid when the pressure above exceeds that from below.

It is in setting the long strings of casing where the heaviest strains come on the equipment, and five sheave travelling blocks are being used to an increasing extent.

A great deal of study has been given to cementing and special accessories have been produced to assist this operation. The practice of cementing strings of casing through perforations just above the oil sand is growing in use. In such cases a basket is used to keep the cement out of the oil sand and to shut off only above the point where the cement is introduced. Reinforcing of the casing by filling between the water and oil strings is also being practised. T. W.

595. Fishing Operations Simplified. Bernard H. Scott. *Oil Field Engineering*, 1928, No. 6, p. 36.

Fishing for tools left behind in the well is now a much easier and less hazardous operation than in former years, owing to the tools being designed to meet practically all needs.

One of the latest is the "pack off" overshot, and one make of this type embodies the utilization of the load springs as cutting knives in case of emergency. The bore of the "pack off" bowl is tapered to form a conical seat for the packing rubber. As the overshot is lowered over a "twist-off" the packing rubber has sufficient clearance to allow the "pack off" bowl to pass down over the "fish" until a tool joint is encountered, the packing being pushed upwards by this action. After the coupling has passed through the rubber, the overshot is raised and the rubber is forced upward tightly around the base of the coupling. The continued upward strain brings the load springs in contact with the base of the packing rubber and forces the springs into the rubber, thus securely locking the tool to the fish in such a manner that circulation can be maintained throughout the entire length of the string as long as a strain is held on the drill pipe. The operation of releasing the fish is a simple matter of loosening a set screw and taking out all four load springs when brought to the surface. An extra set of load springs with high speed tool steel cutting edges at their upper ends are furnished with the overshot, with which the device can be equipped and employed as an outside cutter if so desired.

Another type is the overshot grab, embodying the principle of a wall hook with workable jaws at the lower end. When the grab is lowered into a well, the jaws slide freely through any constricted point, owing to the upper and lower taper on their outer surfaces; but when contact is made with the twist-off, the jaws are immediately closed and locked. The grab is also equipped with a small cutting tool within the body, which may be used as a releasing means if required. The upper end of the device is fitted with a special set of combination socket slips, with the slip teeth cut in the form of a left-hand thread, whereby the tool may be readily disengaged by rotating to the right.

A decided advantage of this device is the opportunity afforded to engage a twist-off that may be lying over beyond the reach of an overshot and still not far enough over in the wall to permit the effective functioning of a wall hook.

A circular knife ripper is another tool very effective in recovering casing. The general design of the conventional type of ripper is followed in the construction, but instead of the rigid horizontal punching blade, a hardened circular cutter is mounted in the sliding block. When the desired ripping point is reached, the drill pipe is rotated to the right, thereby unscrewing the ripper body from the spring collar, after which the drill pipe is raised a short distance, lifting the trip rod up, which action permits the spring actuated latch to move out and securely lock the trip assembly. As the drill pipe is again lowered the upper nut on the trip rod holds the sliding block in position until the inclined plane within the ripper body forces the sliding block outward, driving the circular knife through the casing.

The horizonotal cutter is frequently employed in conjunction with a ripper as a means of cutting cemented casing at horizontal intervals.

Another make of inside cutter is the slip-set type, which employs a set of slips to retain the cutting knives in position while making a cut. When the drill pipe is lowered slowly to the desired cutting point the slip block slides downwards within the slips, forcing the latter outward. As additional weight is applied the knife retaining cone is moved downward and the knives are forced outward. The slip-set cutter is quite positive in operation and also affords a safe means of cutting with practically no risk in so far as "bull-dogging" is concerned, as there is ample passage space between the slips for any steel cuttings to drop freely by the lower part of the body.

The outside cutter is another tool used to recover drill pipe which cannot be pulled or washed free, owing to cavings accumulating about the string, or side-tracked "fish" falling back into the hole. It also replaces the more cumbersome method of stringing up left-hand drill pipe, but has limitations as to what size pipe can be cut; for example, a cutter of the proper diameter to operate in 6¼ in. casing will cut 2½ in. or 3 in. drill pipe, a cutter small enough to operate in 8¼ in. casing will cut 4 in. drill pipe or 4½ in. and 4¾ in. casing, while a cutter of sufficient diameter to cut 5 in. drill pipe cannot be operated in any size smaller casing than 10 in.

The outside cutters are of different types, one of which permits the cut-off string to be raised to the surface with the cutting string, and is known as the "cut and pull" cutter. The operation is quite a safe one, the advantage in time saving, as compared with the older types of casing cutters, is very obvious.

T. W.

596. EUROPEAN OIL WELL EQUIPMENT. L. Steiner. *Oil Field Engineering.* 1928, No. 6, p. 44.

In North American oil field practice, explosion-proof design and installation of electrical equipment is not given extensive consideration, while most European countries have enacted laws and regulations referring to the protection of drilling plants against fire. Open type motors are permitted only when a totally enclosed room is provided for their accommodation, which must be erected at a safe distance from the well, and amply protected against any explosive gases entering therein.

Motors in the European oilfields are mostly of the synchronous three-phase type with continuously contacting brushes. Those with pole-changer and additional delicate adjustment of speed as used in America have not become familiar in Europe. Hydraulic transmission gears and turbine drives, which

have been tried in some oilfields for avoiding the losses occurring at electrical speed variation, have not gained any practical importance.

The so-called "Rapid Percussion Rig" in use in Europe has been developed on the Canadian rig principle. The Canadian rig attains from 40 to 60 blows per minute with a stroke of approximately 16 in., while the Rapid gives 125 blows per minute with a 7 in. to 10 in. stroke, and 150 blows per minute with a 3 in. to 8 in. stroke.

This rig comprises :—

1. A device for lowering and lifting tools.
2. A device for effecting the continuous up and down motion.
3. A device for compensating the tool weight in order to enable the employment of a comparatively small motor for great depth, where the weight of the tools is rather high.
4. The electrical equipment.

The rig has a double armed walking beam pivoted on a central fulcrum, the back end of which is connected to the crank drive and compensating cylinder, the front end being connected either by rod or wire line to a crown walking beam on top of derrick. From the front end of this latter the hoisting rope is suspended, running through a pulley sheave over the crown pulley down to the drum. The drilling tools are suspended from the pulley sheave. By means of this loose pulley sheave the length of the stroke for drilling is reduced by one half. When drilling, the feed of the tool is effected by means of the brake on the hoisting drum. The turning of the bit when drilling is done by one to four men, according to the solidness of the strata and the thickness of the mud.

The rope percussion drilling rig is manufactured in different types for depths up to 5,000 ft. All are on the same principle and differ only in the dimensions and arrangement of walking beams. The larger types have the walking beam in a centralized manner in order to improve the stability, while in the smaller type it is arranged at the side as on the old "Alianca" rig.

The special feature of the rope percussion method is the utilization of the elasticity of the wire rope running over the loose pulley to the walking beam.

When drilling with the rope percussion rig a flushing slush stream is employed, this being forced down under pressure through the hollow rods. When dry drilling—just before the oil strata—the stroke is lengthened, followed by a reduced number of blows. This is done with the assistance of four holes drilled in the lower walking beam.

The compensating cylinder acts as a cushion when lifting up the rods and as a brake when lowering. By this retardation the elasticity of the rod gear and the drilling rope is brought into play and an elastic and powerful blow attained. Generally two-thirds of the rod weight are compensated in this manner.

The output required for drilling work will be about 25–30 k.w. and a motor designed for about 64 k.w. would be sufficient for a depth up to 3,300 ft., considering an overload capacity of 2·5 times of normal load.

When core drilling a rotary table is arranged on the 26 or 33 ft. stage of the derrick. The rotary table is arranged on slide rails in a lengthwise adjustable manner, so as to clear the way when lowering or lifting the tool.

The slush pumps generally used are of the slow running plunger type for belt drive over a reduction gear.

For the production of the compressed air required for operating the compensation cylinder a compressor has to be considered, an air tank being usually arranged between compressor and cylinder. T. W.

See also Abstracts Nos. 506 and 545.

Petroleum Geology.

597. Radiometric Exploration of Oil Deposits. L. N. Bogsiavlensky. U.S.A. Bureau of Mines Information Circular 6,072, p. 13

In another article in this circular, "Experiments with strongly penetrating radiations from the Earth" (*ibid.*, p. 1) a summary of the results of work carried out during the course of several years at the Institute of Practical Geophysics, Leningrad, is given. A gold leaf electroscope shielded with lead covers was used in the experiments.

From this work it may be considered as established that the intensity of penetrating radiations from the earth changes rapidly from point to point, especially in districts rich in radium.

Measurements at the same points repeated at intervals for some years showed no variation of intensity, and demonstrated further that the intensity was independent of meteorological conditions and variations of the emanation content of the air.

By screening the electrometer with lead sheets up to 8 cm. thick it was shown that the main portion of the radiations came from below, and that part of these radiations consisted of hard rays with a penetrating capacity twelve times greater than the rays of radium C.

The radio activity of oil deposits differs from that of the surrounding strata for the following reasons:—

1. Oil as an organic compound possesses an enormous power of absorbing radio-active emanations.

2. The layers underlying the oil bed, which represent repositories of ooze, are richer in radium because of the great absorbing power peculiar to colloids.

These two considerations are sufficient to account for an oil deposit being more radio-active than the shales surrounding it.

For the experiments in the radio-metric exploration of oil deposits the Maikop field was selected in 1924, as its tectonic structure is simple and well understood.

There are two oil horizons in the field, an upper heavy oil, of density 0·920, and a lower light oil, of density 0·840.

The light oil appears to occur in a series of pools, unconnected with one another, and is of greater commercial importance than the heavy oil.

Two parallel lines were taken across the field, and the geological sections on these lines prepared by the geologist, I. M. Gubkin.

Observations were made at six points on one of the lines and at seven points on the other, and, in addition, at two points off the field.

At each of these fifteen points measurements were made at frequent intervals for several days and the figures found to be constant.

Tests with increased thicknesses of the lead screens showed that only strongly penetrating rays were present.

It was found that there was a decided increase in the intensity of the penetrating radiations above the oil deposits.

It was further noticed that the heavy oil caused an increase in intensity to the same extent as the light oil. W. H. F.

598. The Existence of a Salt Dome in the Haut-Rhin Potash Basin of Oligocene Age. M. G. Friedel. *Comptes Rendus*, 184, p. 1028.

It can be considered as proved that a salt dome, even when circular, vertical, and apparently isolated in an unfolded country, is merely a local uplift of a sedimentary bed of salt which was originally horizontal; and that its formation is due partly to the plastic nature of the salt and partly to the

density of this mineral, which is decidedly less than that of the surrounding rocks.

In cases in which for some reason or other the higher surface of the salt in a zone A becomes raised above the surrounding zone B, the force exerted by the overlying beds at B becomes greater than that found at A.

As soon as the force becomes great enough for the salt to behave as a slowly deformable body it begins to flow from B towards A, and tends to reach the surface at A, either raising or piercing the overlying beds.

The mother beds left at depth all round the dome become thinner, while in the dome A a quantity of salt collects which is much greater than that which originally existed in the area of the dome.

One is thus able to perceive that the principle conditions needed for the formation of a dome are: (1) the presence of a salt bed sufficiently large and pure for the factor density to become effective, (2) the burying of this bed under a thickness of sediments sufficient to bring the factor plasticity into play, and lastly the existence of local differences in level in the dome, which may be quite small and due to various causes, but which act as a motive force to set the salt in motion.

No salt domes were previously known in the Haut-Rhin salt basin. Test drilling, however, in combination with an electric survey carried out by Messrs. Schlumberger enabled me to discover the Meienheim dome.

The maximum width of the dome is 1,200 m. and length 8,000 m.; the salt comes to within 150 m. or 180 m. of the surface, though at the surface there is no sign of this great salt uplift.

The local diminution of the electric resistance, first interpreted as a lessening of the thickness of the alluvium, is however certainly due to the saltness of the water in the alluvium and shows that solution is still taking place.

The complete agreement between the results of the electric survey, interpreted in this manner, and of the test-holes, makes it clear that the dome exists and is of the general form mentioned.

It is interesting to notice here the simultaneous existence of the salt dome and the horizontal sedimentary beds of known age, from which it was derived.

Cases in which such circumstances undoubtedly occur are rare, and may serve to eliminate much useless theorising.

According to a report from M. C. Schlumberger, another salt dome has been located by means of an electric survey on the same district.

W. H. F.

599. The Latest Attempt to Find Oil in the Vienna Basin. K. Friedl. *Petroleum*, 23, 6, 1927, pp. 189–272.

The geology of the Vienna Basin is described at some length, and emphasis is laid on the fact that without the help of geophysics it would be extremely difficult to locate favourable oil structures in the area.

A short description of previous geophysical investigations, carried out with the Eötvös balance, is given, and other possible methods—seismic, magnetic, etc.—are discussed.

The method ultimately adopted was the Sundberg electro-magnetic (inductive) method, and it was applied indirectly to find the depth of a salt-water horizon at a series of points.

By mapping the depth of this horizon it is considered that a definite idea of the beclomi structure can be obtained.

Two localities with favourable dome structures were discovered at Schwadorf and Lauzendorf respectively.

W. H. F.

600. The Efficacy of the Magnetic Method for Locating Faults in the Oligocene of Alsace. J. Jung and P. Geoffrey. *Comptes rend.*, 186, p. 1223.

The Oligocene of Alsace is to a great extent covered with quaternary beds, and formerly the stratigraphy and tectonic structure could only be investigated by boring.

Lately the electric method of prospecting has been applied to locate salt domes. It is evident, from the result of the present investigation, that the magnetic method is applicable for locating the lines of certain oligocene faults.

The apparatus used was Brunner-Chasselon magnetic theodolite, usually employed for making absolute measurements of the horizontal component of the magnetic force.

The instrument was modified, in the following manner, to enable it to be used as a variometer.

A magnet was hung from a torsionless fibre at the centre of the instrument, and another magnet fixed to the rotating part of the instrument so that the distance between the two magnets remained constant.

When taking observations the instrument was set up in the magnetic meridian and the upper part rotated through an angle ϕ, until the suspended magnet was at right angles to the fixed magnet.

When used at another station the change in the angle ϕ is proportional to the difference in the horizontal force between the two stations.

Temperature corrections were necessary, and the corrected observations were probably correct to 20 g.

A fault is shown in cross section by a maximum or minimum value of the horizontal disturbing force.

By making several parallel traverse surveys, lines of maxima and minima are found, and these serve to show the lines of faulting.

In an exceptional case a maximum value of 600 g. was measured.

With alluvium 10 M. thick a maximum of 70 g. was obtained.

The contact of the Oligocene and the Bathenian limestone at Lauw was shown by a minimum of 300 g.

The possibility, thus demonstrated, of tracing faults in the Oligocene beneath a cover of alluvium enables the problem of the search for oil in Alsace to be envisaged in a new manner. W. H. F.

601. Venezuelan Oilfields. Ralph A. Liddle. *Oil Field Engineering*, 1928, No. 6, p. 14.

Venezuela, with its 18 oil producing fields, was the third largest producer in 1927, having been exceeded only by U.S.A. and Russia.

Practically all the oil is asphaltic, but at Carano, State of Trujillo, oil with a paraffin base has been discovered.

Earlier observations indicated that the oil bearing horizons were confined to the Cretaceous and Tertiary, but later investigations have greatly modified these opinions.

At El Cantil, State of Monagas, oil and gas escape from crevices in the base of the El Cantil formation. Above this formation about 3,000 ft. of light grey shale is highly petroliferous, and it may be that this horizon, which forms the lower half of the Upper Cretaceous, is the source of the oil which seeps from the El Cantil formation. Asphalt and liquid asphaltic oil, in the Guayuta and Colon formations, both in Eastern and Western Venezuela, suggest convincingly that the oil is indigenous to this horizon of the Upper Cretaceous.

In the Lower Eocene, south-west of Machiques, State of Zulia, there is a small seepage of asphaltic oil, but as no other oil bearing Eocene in that district has been found, though the Eocene is well exposed, and since the Colon shale of the Upper Cretaceous is locally very petroliferous, it probable migrates upwards from this shale and either a Cretaceous or an Eocene source appears equally plausible.

In the Rio de Oro and Rio Tarra fields oil is obtained from the Eocene, but the source is open to discussion. On Rio Cogollo, where the Upper Cretaceous (Colon shale) is highly petroliferous, there is not a trace of oil in the coarse sandstone beds of the Eocene which rest directly on the petroliferous Cretaceous.

North of the old Misoa and Zo fields, State of Zulia, there are great active seepages of asphaltic oil from the Eocene, but the source is unknown. The higher horizons of the Oligocene and Miocene to the south at Mene Grande, are very prolific, and furnish a large production and extensive surface seepages. At various other localities the Oligocene, Miocene and even the Pleistocene carry oil; the Oligocene and Miocene oil is probably indigenous, but the oil in the Pleistocene beds is undoubtedly migratory.

1. The Ambrosio field is situated on Lake Maracaibo and is practically an extension to the north of the La Rosa field. It is apparently on the north end of the uplift, on which are located the Lagunillas, Benitez and La Rosa fields, and production is continuous for nearly 50 kilometres along the strike of a terraced monocline. The oil is thought to be indigenous to the Oligocene, but as tar sands overlay the Oligocene it is possible that migration of oil has taken place and deeper drilling may prove an oil horizon below the Oligocene. The depth to production is between 1,500 and 2,400 ft.

The oil has an asphaltic base, about 24° Bé, the production for 1926 being 1,722,901 barrels and for 1927 272,428 barrels.

2. Concepcion field is 26 kilometres south-west of Maracaibo and comprises a long, narrow, sharply folded anticline trending N.E.-S.W., and is known to be 13 klm. long. The producing horizon is considered to be Upper Oligocene Commercial oil is found between 900 and 1,700 ft., and is of a mixed base. 37° to 38° Bé. The production in 1926 was 838,502 and in 1927 633,782, barrels.

3. El Mene field is in the western part of the District of Buchivacoa. The structure is a small south-west plunging anticline open to east. The sands are the same as those outcropping on crest of the uplift. Fossils indicate their Upper Oligocene age and correlation with oil sands at Hombre Pintado to the east and Quiros to the south. The oil producing zone is composed of thin sand lenses in sandy shale, and lies between 650 and 1,000 ft. Deeper drilling has failed to find older producing beds, so it appears that in this field the production is from the Upper Oligocene.

The oil is of a paraffin or mixed base, averaging 32·5° Bé. The production has increased from 7,500 brls. in 1922 to 2,535,023 brls. in 1927.

4. Guanoco field is situated on the edge of the great Guanoco asphalt lake, and first produced oil in 1913 at 615 ft. The low gravity of the oil and the activity of the asphalt seepages indicate that they are from the same source. The producing horizon is not definitely known, but it is probable that some of the oil is obtained from the Guayuta formation of the Upper Cretaceous. Some or all of it may be derived from the Miocene, which, overlapping the Cretaceous unconformably, is highly petroliferous. The character of the oil is black, asphaltic, averaging 10°–12° Bé. The production is erratic on account of wells being shut in, but in 1927 it was about 614,000 brls.

5. Lagunillas field is situated along the east shore of Lake Maracaibo, on the south end of a terrace or on the west flank of a large uplift. Oil is obtained

from the Oligocene. The base is asphaltic, 17·5° Bé and is notably lower in gravity than oil in the fields to the north. The production in 1926 was 1,218,044 brls. and in 1927 28,789,918 brls.

6. La Paz field is 45 klm. slightly north of west from Maracaibo. Test pits and drilling data suggest that the La Paz structure is a very sharp fold which lifts the Oligocene practically to the surface. The first production was obtained from hard, sandy limestone at a depth of 752 ft., the oil being asphaltic and averaging 25° Bé. The production in 1926 was 734,280 brls. and in 1927 228,910 brls., having been shut down the greater part of 1927 for conservation.

7. La Rosa field is situated on the eastern side of Lake Maracaibo. The structure has not been defined; it may be a monocline or the west flank of a great anticline. Faulting and local steep folding are found in the Miocene and Oligocene, the latter being the producing horizon. The oil sands are soft and under the heavy gas pressure give considerable trouble. The oil is asphaltic, varying from 20°–24° Bé. In 1927 the production was 19,283,208 brls.

8. Mene Grande field is 10 klm. to the east of Lake Maracaibo and is located on the south extension of the southward plunging Misoa nose. The southward plunge brings in Oligocene, Miocene, and later deposits in the Mene Grande area. The largest production is from beds considered to be Oligocene. The sands are soft and carry a great amount of oil under strong gas pressure. The oil is asphaltic, varying from 15° to 19° Bé. The production in 1921 was 982,712 brls. increasing each year, 1927 yielding 9,818,296 brls.

9. Pauji field is 26 klm. from Mene Grande. The structure is very complicated, a fault crossing the field and striking south-west. The age of the producing horizon is thought to be the same as that of the Mene Grande oil bearing beds. A small amount of asphaltic oil was obtained before drilling was suspended.

10. Punta Benitez field is located in Lake Maracaibo 7 klm. south from La Rosa. The field being situated between the Lagunillas field on the south and La Rosa on the north, is doubtless on the same general structure. The producing horizon is between 2,500 and 2,800 ft., the oil averaging from 26° to 28° Bé. The production in 1926 was 135,763 brls., in 1927 1,146,278 brls. During the last few months of 1926 the field was shut down through lack of storage.

11. Quiriquire field is in the State of Monagas. Heavy, asphaltic, black oil from the unconformable overlap of Miocene beds on black shale of the Guayuta formation of Upper Cretaceous age and averages 12° Bé. The oil is similar to that of Guanoco field. The three wells are at present shut in, but a commercial quantity was produced in 1923.

12. Rio de Oro field is 300 klm. S.W. of Maracaibo, and was discovered in 1915. The oil is produced from the lower part of the Third Coal horizon of Lower Middle Eocene age, from a varying depth of 1,085 to 3,150 ft., and is a black asphaltic oil 27° Bé. Development has been retarded by lack of transportation facilities.

13. Rio Tarra field is in the State of Zulia, having been discovered in 1916. Oil occurs in First Coal horizon of Lower Pliocene age; in the Sandy shale (Cubo sands) of Oligocene age; in the Mirador sandstone and in the Third Coal horizon (Tabla sands) both of Eocene age, and in the Colon shales of Upper Cretaceous age. The oil is asphaltic, varying from 23° to 32° Bé. The lower gravity oil is in the upper sands. In the Tabla sands it is from 30° to 32° Bé. Lack of transportation or storage has caused the field to be shut in, but about 25,000 brls. per year were produced during 1925 and 1926.

In 1927 additional drilling was carried out on the Manueles dome, resulting in a commercial producer of 100,000 brls., and this has opened up a new area on the Rio Tarra anticline.

14. Sibucara field is about 7 klm. west of Maracaibo. The producing horizon is in the Upper Oligocene, but only gas has been encountered up to date, the first well in 1926 producing 2,000,000 cub. ft. of gas.

15. Urumaco field is in the State of Falcon. The first oil was encountered at a depth of 3,400 ft. in 1926, the source not being definitely known. The oil has a paraffin base congealing at a high temperature and is between 31° and 33° Bé.

16. The El Mene and El Posone fields are in the State of Falcon, and although for ten years search was made for oil with varying success, the first commercial producer was drilled in 1927. The oil is probably from the Curamichate sands of Upper Oligocene or Lower Miocene age and is of the same character as that of the El Mene field.

17. Hombre Pintado field is situated 30 klm. east of El Mene field. The first commercial well was finished in 1927. The oil is produced from the Upper Oligocene and is of the same gravity as that of the El Mene field. A considerable production must be outlined before full development can take effect as both transport and access are difficult.

18. Totumo field is located in the State of Zulia. Oil was first obtained in 1919 in commercial quantities, in brecciated, quartzitic sandstone and igneous rock at 1,227 ft. The only producing well is now shut in.

Favourable Prospective Structures.

The Limon or Kilometer 24 field, north-east of La Paz, is located on a fairly definite uplift. In 1920 gas, estimated at 10,000,000 to 30,000,000 cu.ft., was encountered at 1920 ft. The horizon apparently exhausted itself and the well was deepened to 2,720 ft. Considering later production and the knowledge of structure, it is very probable that production will be discovered.

At La Danta and at Agua Clara, State of Falcon, good showings of oil and gas have been obtained, but further drilling will be necessary to either prove or condemn these areas.

At Inciarte, near the large asphalt seepages, no drilling has been done for several years, but in view of the recent production in Amana No. 1 well, it is probable that further drilling will be done in the Inciarte area.

The recent completion of Amana No. 1 as a commercial producer, in the State of Zulia, opens up another vast area. T. W.

602. Geophysical Exploration for Ore. Max Mason. *Eng. & Min. J.* New York, Nov. 12 and 19, 1927.

The article contains an account of most of the geophysical methods in common use, with a description of a number of tests carried out by electric methods over models of ore deposits and known ore bodies.

Two points are noteworthy: (1) a simple nomenclature for the different electric methods in use is adopted; (2) the inverse cube law, of the detectability of subterranean bodies with depth, is stated clearly and definitely.

1. Electric methods are classified as:—

(*a*) Self-potential, in which observations are made of the natural electric currents found near sulphide ores (Schlumberger's Spontaneous Polarization Method).

(*b*) Surface potential, in which an artificial, direct or alternating current is made to flow through the earth between two series of metal spikes driven into the ground, and the current distribution on the surface observed.

(*E.g.*, the equipotential line methods due to Schlumberger and Lundberg and Nalhorst.)

(*c*) Inductive method. This method, originally due to Conklin, includes most of the special methods now in use. An alternating magnetic field is created, which penetrates the rocks and soils of the earth nearly as readily as it does the air above. When it encounters a conductor a new magnetic field of the same frequency and general characteristics is emitted by the conductor. This new field radiates from the conductor in all directions in the same general manner as did the original one from the original source. It is called the secondary or induced magnetic field, and is the field to be studied.

To do this the total field has to be measured and an estimate made of what part of it is due to the secondary field.

2. The law of detectability of ores with respect to distance is an inverse cube law, for the Magnetic, Gravitational (torsion balance), Surface Potential and Inductive Methods.

A deep body is theoretically exactly as detectable as a shallow one, provided it occurs on the same relative scale.

Some investigators seem to have been misled upon this topic, and two of the active operators of electrical methods claim in their descriptive pamphlets that an inverse square law is applicable in their methods.

W. H. F.

603. The Discovery, near Hettenschlag, of a Second Salt Dome in the Alsatian Plain. C. and M. Schlumberger. *Comptes rend.*, **186**, 445.

Friedel reported the existence of a salt dome near Meienheim, and mentioned a second dome discovered by means of an electric survey.

A test hole has proved that the second dome exists.

The form of the dome is more circular than that of the Meienheim dome, due apparently to its being of more deeply seated origin.

The Hettenschlag dome is shown by a local uplift 2½ metres high. The salt is thus still rising, and this 2½ metres represents the rise of the surface level since Quaternary times.

A systematic survey of the district, in which the resistance of the soil was measured to a depth of approximately 100 metres, led to the discovery of the Hettenschlag dome. While the resistance of the alluvium was between 300 and 400 ohms. per cubic metre, that of the underlying marl varied between 10 and 20 ohms.

The presence of the dome was shown by a considerable local diminution of the resistance, to be attributed to the smaller thickness of the alluvium and possibly, to a lesser extent, to the increase of the saline constituents of the marl in contact with the salt.

By plotting the measured resistances the approximate shape of the dome was found. Lastly, measurements made above the dome at increasing depths showed, first, that the resistance became less, thus locating the change from badly conducting alluvium to highly conducting marl, and then that it increased considerably at depths where the salt plug, which is an almost perfect non-conductor, was reached.

W. H. F.

604. Porosity Determination by the Acetylene Tetrachloride Method. C. E. Sutton. *Oil Field Eng.*, Sept., 1928, **4**, 3.

The advantages claimed for this method are quickness—the time of the determination being 45 minutes—the small size of sample acquired, and the use of a fairly non-volatile fluid which does not adhere to glass.

Commercial tetrachlorethane is used in a Le Chierlate specific gravity apparatus, and the sample of oil sand to be tested is from three to five cubic centimetres in volume.

The sample is first freed from oil by refluxing with carbon disulphide or petroleum ether, and then dried at from 400° C. –600° C., the sample being rejected if large cracks are noticeable in it. Its volume is then determined. The sample is then removed and powdered finely, after which the last traces of tetrachlorethane are removed. The sample is thus again transferred to the bottle for volume determination. The porosity is determined from the formula :—

$$P = \frac{V_1 - V_2}{V_1} \times 100$$

where P = porosity.
V_1 = volume of solid specimen.
V_2 = volume of sand grains. A. R. S.

Coal and Shale.

605. The Formation of Gaseous and Liquid Hydrocarbons by the Action of Steam and by the Action of Alkali on Semi-Coke at Increased Pressure. F. Fischer and H. Pichler. *Brennstoff-Chem.*, 1928, **9**, 200–206.

With the object of elucidating the mechanism of the Bergius process it has already been shown (Fischer and Frey, *Brennstoff-Chem.*, 1925, **6**, 69; *J. Inst. Petr. Tech.*, 1925, **11**, 160A) that the semi-coke from Rhenish brown coal, when heated with hydrogen under high pressure, yields 30–40 per cent. of its weight of oil, of which the portion boiling below 280° contains 20 per cent. phenols. It then became of interest to determine whether the conversion of semi-coke into gaseous and liquid hydrocarbons could be effected by the use of water instead of hydrogen. The water-gas reaction at high temperatures is: $C+H_2O=CO+H_2$ (I.). At low temperatures the reaction, instead of proceeding according to the equation $C+2\ H_2O=CO_2+2\ H_2$ (II.), might take the course $2\ C+2\ H_2O=CO_2+CH_4$ (III.), or, indeed, higher homologues of methane might also be formed. Previous information on the action of water under pressure on carbon is scanty. Bergius (*Fortschr. anorg. Chem.*, I., **1**, 100) obtains hydrogen and carbon dioxide by the action of water on carbon under pressure at 350° in the presence of thallium chloride. Tropsch (*Abh. Kohle*, 1923, **7**, 107) finds that water and wood charcoal at 500–550° under pressure give carbon dioxide and hydrogen, with some methane. Billwiller (*Fortschr. anorg. Chem.*, II., **1**, 45) obtains hydrogen by heating coal with solutions of caustic alkalis at 350° under pressure. It is now shown that gaseous and (in smaller quantity) liquid hydrocarbons are among the products of the action of water or of aqueous alkalis at 500° C. on various semi-cokes.

Semi-coke from Lohberg "Steinkohle" and from Ilse or Union brown-coal, specially prepared pine-wood charcoal and commercial wood charcoal are employed. These materials are first freed from tar, which would form a possible independent source of hydrocarbons, by heating for 15 hours at 520°.

The semi-coke is then heated in an autoclave at 500° with various quantities of water, the ratio 8 : 1 giving the best results. Gas is evolved in quantity varying with the source of the semi-coke. After three hours the yield is 2,830 c.c. using Ilse brown-coal, 1,700 c.c. from Union brown-coal, 1,170 c.c. from special pine-wood charcoal, 880 c.c. from commercial wood charcoal,

and only 350 c.c. from Lohberg "Steinkohle." The composition of the gas is however, practically constant. Besides 33–43 per cent. carbon dioxide, there are present about 20 per cent. hydrogen and about 40 per cent. methane. The ratio methane : hydrogen corresponds closely with that calculated by von Reinders (*Z. physikal. Chem.*, 1927, **130**, 405) for "ideal" water-gas produced at 380° and atmospheric pressure. The use of more water than 12·5 per cent. leads to a decreased yield of gas, in which the proportions of hydrogen and methane are, respectively, increased and diminished. The use of less water than 12·5 per cent. also gives a lower yield of gas. When the reaction is continued longer than three hours, the yield of gas is increased, but less than in proportion to the time of reaction. This is ascribed to a diminution in the reactivity of the residual semi-coke. The hydrogen and methane contents are, however, decreased and increased respectively by longer heating. Either the reaction: $C+2\ H_2O=CO_2+2\ H_2$ proceeds more rapidly than the (simultaneous) reaction: $2\ C+2\ H_2O=CO_2+CH_4$, or the reaction occurs in stages, methane being formed principally by the interaction of semi-coke with hydrogen produced according to the former equation. The reactivity of the semi-coke remains low when the reaction mixture is reheated after removing the gas produced. No appreciable improvement is effected by adding more water or by drying the residual semi-coke in air or at 350° in a vacuum before reheating. The same sample of partly used semi-coke rapidly gives a large yield of gas (1,940 c.c. in three hours), free from carbon dioxide, when heated with 5-*N* potassium hydroxide solution. This can be repeated on the residual semi-coke, whereby more gas richer in methane and poorer in hydrogen is obtained if more alkali is added, and less gas poorer in methane and richer in hydrogen if the residual gas is merely heated after removing the gas first formed. After 82 per cent. of a particular sample of semi-coke had been used up in this way (26 per cent. going to "gas" and 56 per cent. to carbon dioxide) there was no indication that the reaction could not have been continued even further. In general, the gas obtained using 5-*N* potassiun hydroxide is rich in hydrogen rather than in methane, doubtless owing to the effect of the water so added. The addition of a large excess (900 per cent.) of solid potassium hydroxide gives a large yield of gas free from carbon dioxide and containing up to 44 per cent. methane and 10 per cent. higher gaseous hydrocarbons. A small yield (3–3·5 per cent.) of liquid hydrocarbons, free from phenols, is obtained. Rubidium, potassium and sodium hydroxides in 5-*N* solution produce effects of the same order of magnitude. Ammonia and milk of lime have but slight influence in promoting the reaction. Ammonium carbonate, ammonium sulphide and sodium sulphide in 10-*N* solution produce appreciable quantities of gas, but are less effective than the alkali hydroxides. Iron, nickel, ferric oxide, nickel sulphate, cobalt chloride, nickel hydroxide and cobalt hydroxide are without catalytic or other influence on the reaction between semi-coke and water. W. S. N.

606. Improved Preparation of Cracked Benzine by the Blümner Process (Particularly from Brown-Coal Tar and Shale Oils). Salmony. *Erdöl und Teer*, 1928, **6**, 91–93.

In the Blümner process for cracking brown-coal tar, shale oil, etc., the cracking stock is passed via heat exchanger, preheater, etc., into an autoclave charged with molten lead in which Raschig rings are immersed and in which the oil is rapidly and uniformly heated to 450°. Since every particle of the oil is equally heated cracking is accomplished with a minimum risk of coke formation due to overheating, whereby frequent stopping of the process for cleaning purposes is avoided. The following yields are obtained. (1) From paraffinic or non-asphaltic light oils or (Galician) distillates: 80 per cent.

crude benzine or 70 per cent. refined benzine, 7 per cent. gas and 20 per cent. residue or heavy oil. (2) From asphaltic crude oils (Texas, Ohio), brown-coal tar or light shale oil: about 50–60 per cent. refined benzine, about 12–10 per cent. gas, and 32–24 per cent. asphaltic residue. (3) From brown-coal generator tars, primary tar or heavy shale or lignite tar: about 35–50 per cent. refined benzine, 25–12 per cent. gas and 40–25 per cent. asphaltic, phenolic residue. The benzine contains about 70 per cent. motor spirit and 30 per cent. heavy benzine. W. S. N.

607. Hydrogenation by Pressure of an Eocene Peat. J. Varga. *Brenn.-Chem.*, 1928, **9**, 277–282.

Experiments are described in which a pulverised Eocene peat (giving 9·05 per cent. yield of tar and 1·1 per cent. water vapour) was submitted without the addition of tar to hydrogenation by pressure. The powder was heated in rotating autoclaves with hydrogen containing 1·6 per cent. carbon monoxide and 0·9 per cent. nitrogen. The effects of temperature, of the duration of heat, of added iron oxide, of the initial pressure and of the relative quantities of hydrogen applied were observed. Experiments were carried out with 500, 400, and 300 gms. coal respectively in a 3·73 litre bomb at 450–480° and under initial pressures of 75, 100, and 125 atmospheres respectively. The period of heating was 72–77 minutes to the reaction temperature, and one minute, one hour or three hours at the desired experimental temperature.

Effect of Iron Oxide.

An addition of 15 per cent. iron oxide was mixed with the pulverised coal, since the latter had a high sulphur content of 3·98 per cent. The oxide was found to reduce the sulphur content (the greater the quantity of iron oxide the less the hydrogen sulphide content of the gas), and was capable of producing a gas free from H_2S. It also had a catalytic influence—namely, causing a decrease in the temperature of hydrogen displacement, an increase of oil yield and of H_2 consumption.

Influence of Period of Heating, of the Temperature and of the Relative Quantities of Hydrogen.

These factors influenced not only the quantity but also the quality of the oil produced. In the Bergius liquefaction of coal under H_2 pressure at high temperatures, hydrogenation and coking occur and at the optimum temperature the equilibrium is displaced in the direction of hydrogenation. Each reaction factor can influence the equilibrium.

The quantity, specific gravity and composition of the gas formed is dependent on the reaction temperature, on the period of heating and on the applied pressure. It has been shown that the longer the hydrogen is allowed to react with the coal or the higher the reaction temperature, heavier and substances higher in calorific value are produced.

The coal could be converted into 16·8–57·9 per cent. yields of oil (calculated from pure coal) with specific gravities ranging from 1·002–1·066. The maximum yields of oil was obtained when the heating lasted one minute and on further heating the oil was decomposed; the best initial pressure was 125 atmospheres under which pressure the coal was primarily converted into high-boiling products and then these were decomposed into lighter, lower-boiling oils.

In experiments with 500 gms. coal at 480° C. and at 100 or 125 atmospheres initial pressure, 52 per cent. or 51·8 per cent. oil yields were obtained respectively. The maximum oil yield of 57·9 per cent. was obtained with 300 gms. of coal at 470° and at 100 atmospheres initial pressure. W. S. C.

608. A Process of Converting Coal into Hydrocarbons. A. Gaertner. E.P. 266,311, 1928.

A mixture of pulverised coal, steam and finely powdered iron or other metal capable of producing hydrogen by reduction of steam is injected under pressure into a reaction chamber from which the hydrocarbons and solid products are subsequently separately removed. The steam, which may be mixed with carbon monoxide, is used at a temperature exceeding 400° C., and the coal may be introduced in a preheated condition. Lime or other alkali may also be added to fix the resultant carbon dioxide, the excess of which is separated from the gaseous products by solution under pressure in water. The apparatus consists of a chamber containing a bell-shaped appliance into which the steam, coal, etc., are injected from the bottom at an elevated pressure—e.g., 25 to 30 atmospheres. The solid reaction products fall out of the bell whilst the vapours pass through a dust separator placed between the bell and the chamber and are taken to coolers and condensers.

H. G. S.

609. Improvements in the Manufacture and Production of Valuable Hydrocarbons and Derivatives Thereof from Coal, Tars, Mineral Oils and the Like. I. G. Farbenind A.-G. E.P. 272,190, 1928.

The hydrogenation of coal tars, mineral oils, etc., under high pressures and at elevated temperatures is effected by exposing the initial materials in a coherent thin layer to the action of hydrogen, the materials being conveyed mechanically through the reaction chamber. Metallic surfaces of large superficial area are preferably used and solid materials like magnesia, diatomaceous earth and the like may be added to increase the reacting area of the raw materials, with which catalysts like molybdic acid may also be used.

H. G. S.

610. Improvements in the Production of Low-Boiling Point Oils by Thermal Treatment of Coals, Oils and the Like. I. G. Farbenind A.-G. E.P. 272,194, 1928.

The process consists in the heat treatment of tars, oils, suspensions of coal, etc., under pressure in the presence of catalysts and passing the resultant products in the vapour state at substantially higher temperatures over catalysts Either or both stages of the treatment may be carried out in presence of inert gases or of hydrogen, etc. The hot zone should not consist of materials which give rise to coke deposition and methane formation. H. G. S.

611. An Improved Process for the Destructive Hydrogenation of Coal, Tars, Minerals and the Like. I. G. Farbenind. A.-G. E.P. 272,539, July 19, 1928.

It is important that the materials when subjected to destructive hydrogenation be brought into contact with the hydrogen containing gas. This condition may be attained if the gases are passed through a porous mass upon which the materials rest. The porous mass may consist of fragments of glass quartz, earthenware, metal, charcoal, or the like, and is of such a nature that the material does not penetrate. When, for example, middle oils are converted into benzine the porosity of the mass and the velocity of the gases may be so arranged that the oil is converted into foam. The reaction may be carried out in a variety of ways, with or without catalysts and under any convenient pressure.

As an example, intermediate oil may be fed continuously on to a porous layer or plate composed of sintered glass powder arranged in an apparatus

the hot portions of which are lined with an alloy prepared from about 10 per cent. chromium, 2 per cent. molybdenum, 10 per cent. cobalt, and 75 per cent. iron by fusion in vacuo. Hydrogen is admitted through the porous layer from below and according to the rate of flow an intense whirl or vertical movement is produced. If the reaction materials are heated to about 550° C., an almost quantitative yield of oil is produced having a boiling point 30° C. lower than the original oil and containing 25 per cent. benzine. This result may be considerably increased by the use of catalysts and the application of pressure. After separation of the benzine the residue may be treated again in the same manner. R. P.

612. Improvements in the Manufacture and Production of Valuable Liquid Products by the Destructive Hydrogenation of Coal, Tars, Mineral Oils and the Like. I. G. Farbenind. A.-G. E.P. 273,712, 1928.

The raw materials—e.g., coal, mineral oils, tars, pitches, bitumens, asphaltums or their distillation and conversion products—are first treated under high pressure with hydrogen in the presence of cracking or splitting catalysts and then with catalysts which facilitate hydrogenation. The operation is conducted with a current of hydrogenating gas and with a large excess of hydrogen. The cracking catalysts specified are the elements of groups three and four of the periodic system, copper-iron mixtures or compounds of the same, active charcoal, active silica, hydrosilicates, alumina, magnesia and the like, whilst the hydrogenating contacts comprise cobalt, molybdenum, tungsten, etc., or their compounds.

Example 1. A brown-coal tar fraction is injected into a vertical reaction vessel lined with aluminium and whilst in the liquid state at 450° C. and 200 atmospheric pressure, is made to flow in counter-current with an excess of hydrogen in the presence of active charcoal. The highly unsaturated products are then treated in the vapour state at 450° C. with a cobalt catalyst and thereby hydrogenated. The liquid product obtained by cooling the vapours is a liquid, 90 per cent. of which is practically saturated benzine, and is suitable for use as engine fuel without refining. *Example* 2 deals with the treatment of a mixture of coal and a viscous oil. H. G. S.

613. Destructive Hydrogenation of Carbonaceous Materials. I. G. Farbenind. A.-G. E.P. 275,662, August 8, 1927.

This is an improvement of the invention described in specifications Nos. 268,796 and 271,451, which consists in carrying out destructive hydrogenation of carbonaceous materials under pressures of at least 50 atmospheres with a large excess of hydrogen in the form of a stream in the presence of catalysts. Formation of carbon monoxide and methane as well as the deposition of carbon and other undesirable by-reactions are avoided if the parts of the apparatus coming in contact with the reacting material consists of or is coated with alloys containing cobalt, molybdenum, nickel, boron, arsenic, antimony, silicon, bismuth, phosphorus or selenium. When the initial materials are free from sulphur, copper or alloys thereof may be employed. Such metals and alloys are also very suitable in which the boundaries of the crystal grains are substantially free from impurities as when prepared by fusion in vacuo. When prepared in this way these materials in the form of sheets or wires may also be employed as catalysts of high stability. H. S. G.

614. A Process for the Manufacture of Motor Fuels. I. G. Farbenind. A.-G. E.P. 281,247. August 2, 1928.

By the destructive hydrogenation of brown-coal, motor fuels of low-boiling point are obtained which are liable to knock. Knocking is due to the cracked

products of the bitumen of the coal and the primary products of conversion, such as paraffin wax, yielding a spirit which is inclined to knock. However, excellent motor fuels of anti-knock value may be obtained from brown-coal or highly bituminous coal by working under such conditions that the bitumen itself or the primary conversion products are not decomposed, whilst the other constituents of the coal are converted into low-boiling products. The term bitumen here refers to all the constituents of the coal which can be extracted by benzine.

An advantageous method of eliminating the bitumen, prior to or during the intermediate stage of the treatment, is, for example, by extracting the material with benzine or similar solvents at atmospheric or elevated pressure. The resulting coal is then treated with hydrogen at a high pressure and temperature, with or without catalysts and a satisfactory anti-knock fuel obtained.

This method may be modified by removing the bitumen or paraffin wax in an intermediate stage of treatment. The middle oils which contain bitumen and paraffin wax are converted into spirit by treatment with hydrogen under very mild conditions so that the bitumen and paraffin undergo only slight modification, then recovering them in a pure state by strongly cooling. For example, brown-coal containing 8 to 10 per cent. of bitumen when treated with hydrogen at a high temperature yields a spirit with a tendency to knock in an engine at a 1 : 5 compression ratio, but after extraction with benzine the product does not knock, under the same engine conditions.

The coal may be converted largely into middle oils which are cooled to a low temperature and the bitumen and the paraffin removed in the solid form. The remainder is then converted into motor fuel. Again, with the aid of catalysts the middle oils may be converted to the extent of 10 to 20 per cent. and the bitumen and paraffin wax extracted from the product by cooling, then further conversion may be carried out. This last method has the advantage that the extensively purified paraffin wax is obtained as a by product. R. P.

615. Production of [Fuel] Gases. J. Y. Johnson. E.P. 293,413. April 2, 1927.

An improvement relating to E.P. 214,544 is described, according to which fuel gas is produced by blowing a gas adapted to produce fuel gas through small-sized ignited coal or carbonaceous material with sufficient speed to establish a strong agitation of the material so that it presents the appearance of a boiling liquid. The gasifying agent is now admitted through very small vertically-directed openings in a horizontal cooled plate or grate, which acts as a support for the solid fuel and which may be a travelling grate. It is an advantage to be able to close the openings independently, whereby the gasifying agent may be admitted through only a portion of the support, preferably the centre. W. S. N.

616. Improvements in the Manufacture and Production of Hydrocarbons. I. G. Farbenind. A.-G. E.P. 293,887. 1928.

Liquid hydrocarbons obtained by the destructive hydrogenation of coals, tars, mineral oils or their fractions are submitted to dehydrogenation in the presence of catalysts at temperatures exceeding 300° C. and preferably under elevated pressure. Oxides of the metals of the sixth group alone or mixed with each other, or with oxides of the elements of the third and fourth groups are specifically mentioned. The catalysts may be pre-treated with hydrogen. Substantial yields of aromatic hydrocarbons with evolution of hydrogen are claimed. H. G. S.

617. An Improved Method of and Means for Treating Oil-Bearing Shales and Kindred Oil-Bearing Minerals. N. H. Freeman. E.P. 291,475. 1928.

The process is an improvement of E.P. 248,014 and consists in heating shales or other oil-bearing minerals until their contained hydrocarbons become soluble, thereafter extracting the heated mass with an entraining agent. The latter may be the oil produced in the first stage of the treatment or alternatively superheated steam, oil vapour or like gaseous medium may be employed as the entraining and distilling medium. The raw material is first raised to critical temperature in a retort and is then transferred to a rotating extraction vessel to which the gaseous entraining agent is admitted, the oil being taken to a condenser and the solid residue removed by means of extraction gear. H. G. S.

See also Abstracts Nos. 485, 495 and 518.

ABSTRACTS.

Arranged by W. H. Thomas, A.R.S.M.

Abstractors.—S. F. Birch, Ph.D., D.I.C., W. S. Clarke, B.Sc., R. W. L. Clarke, B.Sc., S. W. Cooke, B.Sc., H. S. Garlick, B.Sc., A.I.C., W. N. Hoyte, B.A., W. Littlejohn, W. S. Norris, Ph.D., D.I.C., G. Sell, H. G. Shatwell, Ph.D., M.Sc., R. Stansfield, A.M.Inst.C.E., A. R. Stark, B.Sc., A.I.C., H. E. Tester, B.Sc., W. H. Thomas, A.R.S.M., T. Wiskin.

General.

618. The Petroleum Industry for 1927/28. N. Tolstonogov. *The Oil Bulletin*, 1928, No. 20, p. 2.

The production of petroleum from the three main oilfields reached in 1927/28 the record figure of 11,278·4 thousand tons. In comparison with the previous year the increase was 11·8 per cent. This is a significant development in both the larger fields, Grosny in this period having doubled its record figure of 1917. There was an insignificant decrease on the Emba field, owing to the exhaustion of the Dossor area and the unjustified expectations of the new wells at Makata.

The increased production in Grosny and Baku was due almost exclusively to the growth of "fountain" areas—New Grosny and Turachani.

Both Turachani and New Grosny gave an unprecedented proportion of paraffin petroleum which was reflected in a liquid fuel of higher pour point.

The production of gas in Baku and Grosny was 268 thousand tons, an increase of 9·8 per cent. over the previous year.

A significant set-back in prospective drilling was noticeable on both exploitation and new areas—approximately 53 thousand tons, a decrease of 25 per cent. as compared with the previous year.

8,698 thousand tons of oil, 24 per cent. more than last year, were delivered to the refineries. As a result of extensions and better utilisation of both old-fashioned and new installations, distilling operations overtook the development of production—only 23 per cent. of the total production remained unrefined as against 31 per cent. the previous year. The question of distillation was most acute with Azneft, where 31 per cent. of untreated crude oil remained. Even here an improvement is noticed—41 per cent. of crude having remained undistilled the previous year. The stabilisation of the output of benzine by Azneft should be noted. In Grosny the output of benzine rose from 16·8 per cent. to 17·3 per cent. This insignificant increase is explained by the increased output of light benzine at the expense of heavy.

The sales of benzine increased by 13·5 per cent. while those of kerosine increased by 15·3 per cent. owing to an increased consumption and a stronger demand by tractor proprietors. Lubricant and special product sales also increased by 14·8 per cent.

Export reached a record figure of 2,727 thousand tons—34 per cent. in excess of the previous year. All groups of oil products were exported in larger quantities, the increase on benzine being 26·6 per cent. The realisation of kerosine was augmented 1½ times.

Amongst the importers of Russian oil products the first place is occupied by Italy, and although its participation in exports decreased from 23·4 per cent. to 18·1 per cent. the total exported increased by 3 per cent. on the preceding year.

The second place belongs to England, where the imports, for the accounting period, grew by nearly 2 per cent.

France retains third place and her participation in Russian oil exports fell to 13 per cent. against 18·9 in the previous year.

The disposal of fuel to foreign steamers was almost doubled.

In general the total realised on both the interior and foreign markets was 9,424 thousand tons, 1535 thousand tons more than the previous year, with an increase in the production of oil of 1193 thousand tons. T. W.

619. Extension of Paraffin Works: General News. *The Oil Bulletin*. 1928, No. 19, p. 12.

The large demand for kerosine has compelled Grosneft to enlarge their works, at present producing 350–400 tons monthly. It is intended to increase the output to 1500–1600 tons monthly by 1932/33.

Two new tubular batteries are now being created, with a capacity of 500 tons mazout each, to distil paraffin mazout, as the present 14-still battery will not be able to supply sufficient kerosine distillate to the refinery in its enhanced capacity. T. W.

620. The Caspian Sea—Moscow Pipe-line. Interview with I. N. Strigeoff. *The Oil Bulletin*, 1928, No. 19, p. 10.

One of the chief problems of the Russian oil industry is the reconstruction of the geographical distribution of petroleum, its distillation and transport.

Petroleum is obtained and refined in remote districts, the resultant products being transported long distances—each in a special vessel, thus enhancing costs.

In America, refineries are concentrated in the main consuming districts and from these refineries the products are delivered by means of short pipe-lines, railroad or motor wagons.

In preparing a five years' programme the "district distillation" of oil and disposal of its products are to be considered. Several main districts have been noted, in each of which a refinery should be erected. The first is the Central Industrial District with Moscow as its centre. Here there should be a large refinery, and consequently a pipe-line should be laid to it.

As sea transport is cheaper than that through a pipe-line, it would not be beneficial to lay the latter from Baku along the Caspian Sea coast, but to commence the pipe-line to the north of this sea and avoid transport up the River Volga to Moscow as river transport is dearer than by a pipe-line.

The first problem in the projected Caspian Sea—Moscow pipe-line is to find a place on the north of the Caspian Sea accessible to ships and requiring the minimum expenditure in deepening and equipping a port.

The pipe-line should be designed to pump oil through to the end without considering deliveries at any intervening point.

Owing to financial strictures, a 10-in. pipe-line with a pumping capacity of 1·7 million tons of crude oil yearly should at present only be considered. Afterwards another similar one could be laid parallel to it.

In conformity with this pipe-line the projected refinery should have a through-put capacity of 1·7 million tons yearly, but provision should be made for future extension. It would be desirable to erect the refinery on the River Moscow below the town, but choice of other places is not excluded.

T. W.

621. Treating Plant Emulsions—Some Theory and Practice. L. S. Catlin. *The Refiner*, 1928. 58.6.7.

The emulsifying agent is not usually easy to identify and the amount of it may be very small compared to the amounts of liquids. It is in most cases a colloid, that is, not completely soluble in either water or oil, and yet not sufficiently insoluble to precipitate, and hence tends to collect at the interface between oil and water. This colloid material has a definite preference for one of the liquids, or is more readily wetted by one than the other, usually considered to be the continuous phase.

During refining, emulsions frequently occur in the doctor solutions which are of the type in which the oil is the disperse phase. Here the emulsifying agent is probably lead mercaptide, which accumulates in the doctor solution by continued circulation. The method of prevention is either the use of more sulphur, or better distribution of the same amount of sulphur. Once the emulsion is formed, however, the addition of sulphur fails to break it. Two methods of breaking this emulsion are given, firstly, by the addition of a strong solution of caustic soda in an amount equal to one-third to one-half the bulk of the emulsion, and followed by the addition of an equal amount of water, and, secondly, by the addition of spent caustic soda from the H_2S wash and heating till the oil and caustic separate readily. The lead sulphide formed settles readily to the bottom. The oil is skimmed off, the caustic soda pumped out, and the lead sulphide sold as such or restored by air blowing while heating with fresh caustic.

Acid-sludge emulsions furnish a good example of the "water in oil" type. A general description of the method of breaking these is given as follows: The emulsion is collected in the regular separators used for washing acid sludge. It should be allowed to stand for 24 hours while heating with a closed steam coil in order to settle as much as possible of the acid. After the latter is drawn off, spent caustic solution is added, a little at a time, while agitating with air or open steam. Tests for acidity are made with any suitable indicator. The emulsion usually breaks when the action is slightly on the acid side of the neutral point. S. W. C.

622. Process for Resolving Emulsions of Tar or Oil. H. B. Robinson and D. W. Parkes. U.S.P. 1,687,314. Oct. 9, 1928.

This invention relates to the separation and recovery of tars or oils from aqueous emulsions or suspensions.

A quantity of finely divided powder is brought into intimate contact with the aqueous emulsion of tar or oil at such a temperature that one of the phases of the emulsion is agglomerated into a thick mass. The powder is preferably added to the emulsion, and the temperature is so controlled that the powder agglomerates the phase to be removed. By suitably altering the conditions of the process first one and then another of the phases may be caused to separate readily on top of the emulsion. Emulsions containing from 30-35 per cent. of water are found to be most suitable for treatment, being treated with the demulsifying agent with heating to about 100°C., when the oil or tar separates out rapidly. Emulsions containing over 40 per cent. of water may be treated by the same method, but are best treated by, first, blending with another emulsion until the water content lies between 30 and 35 per cent. The finely divided powder used in the process is either granite or slate powder, which is easily wetted by water and, moreover, possesses a large superficial area. Powders prepared by chemical methods are found to be quite unsatisfactory. A demulsifying powder which has been used in this process may be recovered and used again by drying at 100°C. until the water content falls below 0·25 per cent. H. E. T.

See also Abstracts Nos. 662 and 631.

Chemistry of Petroleum.

623. The Relative Rates of Bromination of the Olefines. H. S. Davis. *J. Amer. Chem. Soc.*, 1928, **50**, 2769-1780.

Contrary to general belief, the bromination of ethylene in carbon tetrachloride proceeds at a measurable pace, and if water and bright light are excluded the reaction requires a considerable time for completion. The rate of bromination is, however, enormously increased by bright light. The speed of the reaction in the dark varies with the moisture present, oxygen and hydrogen bromide having very little effect. The temperature also exerts some effect as it was found that the rate of bromination in the dark increases progressively as the temperature is lowered from 25° to 0° C. The explanation offered is that the dark bromination occurs mainly through a bromine hydrate, the conversion of which increases as the temperature is lowered. The rates at which olefines brominate in the dark in carbon tetrachloride solution vary considerably, although it was found that the bromination of propylene was considerably faster at 0° C. than at 25° C., the reactions with trimethylethylene and with di-*iso*-butylene were little affected by temperature changes. As hydrogen bromide was given off in both of these reactions, it is assumed that although this substance exerts no effect on the bromination of the lower members, it influences the reaction with the higher olefines. The effect of light is to increase the speed of the reactions but at the same time to decrease the relative differences between them. The author suggests that a study of the action of halogens on olefines may clear up many of the problems connected with the photochemical reactions of hydrogen and halogens. The methods used to follow the rate of bromination were colour comparison using a standard bromine solution and titration of the bromine. Details of both methods are given.

S. F. B.

624. The Synthesis of Aromatic Hydrocarbons from Methane at Ordinary Pressure and without Catalysts. F. Fischer. *Brenn.-Chemie*, 1928, **9**, 309-316.

Bone and Coward's researches on the decomposition of methane show that products other than carbon and hydrogen can be formed according to the experimental conditions. The results are given of five experiments in which methane was heated for various periods of time in a closed porcelain tube at about 1,000 °. Acetylene or unsaturated hydrocarbons were only produced when the methane concentration exceeded 60 per cent., and with a further decrease in concentration of CH_4 the gas was decomposed directly into its elements. The velocity of flow, *i.e.*, the period of heating, was found to be of even greater importance than the temperature of heating. Numerous experiments have been performed on these lines to find the effect of duration of heating, velocity of cooling, and the influence of catalysts.

Berlin hard porcelain and quartz tubes were the most suitable for these experiments. The methane was produced from aluminium carbide and consisted of 83 per cent. CH_4, 9·2 per cent. H_2 and the rest N_2. At least 50 per cent. of the light oil obtained consisted of benzol and the remainder of the liquid hydrocarbons was principally aromatic. Decomposition occurred according to the equation $CH_4 = C + 2H_2$ and there was an optimum temperature for the decomposition of CH_4 and another for the combination of decomposition products.

A review of various patents—namely, Spindler's Patent 255,493, the Compagnie de Bethune's Patent 615,972, an English Patent 258,608, and the French Patent 610,543 of Le Pétrole Synthetique—in which catalysts are

utilised under pressure, reveals the fact that for the production of liquid hydrocarbons under high pressure, catalysts appear to be essential. However, the application of catalysts *as well as* pressure is not necessary in the production of benzol hydrocarbons, and it is not yet known whether better yields will be obtained under these conditions than are obtainable at ordinary pressure in a single process.

W. S. C.

625. Partial Oxidation of Methane and Ethane in the Presence of Catalysts. T. E. Layng and Roy Soukup. *Ind. Eng. Chem.*, 1908, **20**, 1052-1055.

The catalytic partial oxidation of various mixtures of methane and ethane with oxygen has been studied by the dynamic method at atmospheric pressure within the temperature range of 100° and 700° C. Some experiments were also made with natural gas under similar conditions.

For the production of alcoholic or aldehydic intermediates, catalysts of copper and silver or their oxides, activated charcoal, platinum oxide and barium peroxide, were found to be unsatisfactory owing to the poor yield and the other products formed. On the other hand, small quantities of nitrogen peroxide when added to the hydrocarbon-oxygen mixtures increased the oxidation materially when passed through heated capillary tubes. Yields of oxygenated derivatives varying from 15 to 30 per cent. by volume of the hydrocarbon consumed were obtained. As much as 38 per cent. of the hydrocarbon has been oxidised in one cycle. Auxiliary catalysts used in conjunction with nitrogen peroxide activated the decomposition of intermediate oxidation products without increasing the quantity of hydrocarbon decomposed.

Methyl nitrite has been found to exert a promoting action in the partial oxidation of hydrocarbons. The authors offer an explanation of the effect of nitrogen peroxide and methyl nitrite as oxidation promoters.

S. F. B.

626. A Study of Auto-Ignition Temperatures. Part II. Pure Compounds. H. J. Masson and W. F. Hamilton. *Ind. Eng. Chem.*, 1928, **20**, 813-816.

The investigation described in the previous paper (*Ind. Eng. Chem.*, 1927, **19**, 1335) has been continued. The auto-ignition temperatures of an additional number of pure organic compounds have been determined in air and at ordinary pressures. A consideration of the results obtained emphasises several interesting facts, for instance that substances of widely different structures and compositions may have the same auto-ignition temperature, while those having similar or related structures and compositions ignite at widely differing temperatures. The conclusion of previous investigators that increase in molecular weight or complexity decreases the auto-ignition temperature is not borne out by the present work. The effects of surface temperature, sensitivity and heat capacity were also examined and it was found that results are only reproducible when the plate on to which the substance is dropped is at equilibrium with the air inside the furnace, and that, if the temperature of the furnace is raised or lowered, deviations occur due to the time required for heat to pass to or from the plate. The time required for the flash to take place after the drop strikes the plate is a complex function of several factors such as vapour pressure, heat capacity and transference, rate of diffusion, etc. The surface of the plate is, after the composition of the substance, the most important factor in determining the auto-ignition temperature. This is due to the fact that

two forms of combustion take place: (1) gaseous combustion (indicated by the flash) and (2) surface combustion (flameless combustion at the surface). If the surface is of low catalytic activity, all the combustion is in the gaseous phase, but if the activity of the surface is high, no flash is seen as the combustion takes place at the surface. The authors have found that the activity of some surfaces alters with use, the activity increasing with the result that the auto-ignition temperature increases. In determining auto-ignition temperatures it is therefore essential to test the surface activity from time to time by the use of a standard, *e.g.*, benzene. S. F. B.

See also Abstract No. 627.

Crude Oil.

627. Chemical Composition of Rumanian Crude Oils According to Their Origin and Geological Occurrence. N. Danaila and (Miss) V. Stoenescu. Petr. Zeit., 1928, **24**, 1303-1309.

Rumanian petroleum is found in the Oligocene at Bustenari (Prahova), Moinesti, Zemes and Solont (Bacau), in the Miocene at Moreni, Bustenari, Câmpina, Urdresti, Filipesti-de-Padure, Ceptura and Runcu (Prahova), Ochiuri (Dâmbovita), and Arbanasi (Buzau), and in the Dacian formation at Moreni and Baicoi (Prahova), and Gura Venitei (Dâmbovita). Sp. gr., flash-point, volume of distillate to 150° and between 150° and 300° are given for 18 crude oils from the above sources. Each oil is separated into fractions b.p. up to 145° and 145-300°, the former fraction being redistilled, using a three-bulb Lebel-Henning column to give fractions of b.p. 65-95, 95-115, 115-145°. These and the original 145-300° fraction are dried over calcium chloride and finally distilled over sodium. The composition (% of aromatics, olefines, *cyclo*paraffins and paraffins) of these fractions is then determined. The composition varies from one formation to another, even in the same locality. It also varies within each formation. There is no relationship between the composition and the geological origin of the oils. The following classification is based on the content of light aromatic hydrocarbons: (*a*) crude oils from the Miocene at Moreni and the Dacian at Baicoin 4%, (*b*) oils from the Dacian at Moreni and Gura Venitei and from the Miocene at Arbanasi 2½%, (*c*) from the Oligocene at Solont, Moinesti, Zemes and Bustenari and from the Miocene of Bustenari-Gropi and Runcu 1-1½% of volatile aromatic hydrocarbons. W. S. N.

628. Fractional Distillation of Petroleum. J. E. Bell. U.S.P. 1,683,150. Sept. 4, 1928.

This invention relates to an improved method for the fractional distillation of petroleum, particularly to a process in which fractions of intermediate boiling point are separated from the oil. The crude oil is subjected to a vapourising operation in which the gasoline and gas oil fractions are first removed. The heavier vapours are subjected to a regulated refluxing operation in a reflux tower, a light distillate being introduced through the upper end of the tower. This distillate will be re-evaporated in the upper part of the tower, and cause condensation of the heavier rising vapours, including the vapours of the desired fraction. The condensed vapours will flow downwardly in the tower and will in turn condense heavier vapours and be re-evaporated in lower parts of the tower. This condensing and re-evaporating process will take place throughout the height of the tower, so that the heaviest vapours will be returned as liquid to the still, while the lightest vapours escape from the top of the tower to a condenser. The vapours of the required fractions, in excess of the amounts required for

alternate condensation and re-evaporation in the tower, are removed at intermediate points of the tower. These vapours will also extend both above and below the points at which the fractions are drawn off, and will serve to vaporise the lighter constituents and condense the heavier constituents respectively.

In this manner lubricating oil fractions of any desired boiling point range and relatively free from constituents of higher or lower boiling point can be obtained. H. E. T.

629. Method of Distillation of Petroleum. E. W. Isom. U.S.P. 1,683,135, Sept. 4, 1928.

An improved method and apparatus are described, suitable for the distillation of crude oil, in order to produce gasoline, gas oil and coke. To carry out the distillation, the still is charged until about one-third full of crude oil, and the vapours distilling off are subjected to a refluxing operation in a refluxing tower until the desired light distillate is removed from the still charge. At this point, crude oil is fed in through the top of the reflux tower, at a rate sufficient to give a maximum reflux ratio with the vapours from the still. The heating is continued with proper regulation of the crude oil feed, in order to obtain the desired end point gasoline. When the still contains a charge of oil sufficient to fill it to a predetermined level, the oil feed into the reflux tower is shut off, and the heating continued until the vapour temperature at the top of the reflux tower is 450° F. The still is now put in direct communication with a condenser and without any refluxing operation of the charge run down to coke.

It is claimed that the maximum yield of gasoline from a crude oil can be obtained by this process during one operation, the distillation being then continued in the same still for the production of a gas oil fraction and coke. H. E. T.

630. Distillation of Crude Petroleum. E. W. Isom and E. C. Herthel. U.S.P. 1,683,193, Sept. 4, 1928.

The patent describes an improved method of producing gasoline from crude petroleum, whereby its natural gasoline content, together with an additional quantity of gasoline produced by mild cracking, is recovered in a single operation.

The process consists in maintaining a body of the oil in a vaporising zone together with a thin bed of absorbent or filtering material entirely within this body of oil and extending entirely across it. The oil is circulated from beneath the filtering bed through heating tubes and back to the oil above the absorbent bed; the oil is thus subjected to mild cracking temperatures under relatively low atmospheric pressure. The gasoline vapours from the vaporising zone are subjected to a refluxing operation, while at the same time crude petroleum is introduced into the reflux vapours. In this manner the gasoline content is recovered, while the reflux condensate is returned to the body of oil in the vaporising zone. The residual oil, after removal of the gasoline, is continuously run off from beneath the filtering bed. H. E. T.

See also Abstracts Nos. 622 and 672.

Natural Gas.

631. Gas Well on the Vosnisensky Field. *The Oil Bulletin*, 1928, No. 20, p. 12.

A powerful blow out of gas occured in Well No. 12 on the Vosnisensky oilfield from a depth of 688 m., lifting up the drilling tools (weighing about

two tons). The blow-out was short-lived as the well soon became plugged with sand. While cleaning out the plugging on September 18 a strong gas fountain broke out, throwing out stones and sand with a small quantity of oil. At present the fountain is suppressed and preparations are being hurriedly made for capping. The strata from which the fountain took place corresponds to XVI horizon of the Old Grosny district. T. W.

632. Process for Recovery of Vapours. R. T. Osborn, Assr. to Standard Oil Co., California. U.S.P. 1,685,501, Sept. 25, 1928.

Light petroleum vapours which are normally not condensed under atmospheric conditions of temperature and pressure may be readily recovered by passing such vapours into the vapours of relatively higher boiling-point fractions and condensing the resultant mixture. A preferred embodiment of the invention consists of a still, from which light petroleum vapours are distilled off through a vapour line to a condenser. A trap permits only liquid to pass off from the condenser, a vent pipe being provided for uncondensed vapours. A second still operates simultaneously with the first but produces vapours of relatively higher boiling-point which pass via a vapour line to its condenser. The vent pipe of the first still is connected to the vapour line of the second still, whereby the escaping gases from the first still are introduced and comingle with the relatively higher boiling point vapours of the second still, and pass together through the condenser where substantially all the mixed vapours are condensed. H. S. G.

633. Process of Separating Gas Mixtures. A. B. Ray, Assr. to Carbide and Carbon Chemicals Corporation, New York. U.S.P. 1,685,883, Oct. 2, 1928.

A process is described of treating a gas mixture for the recovery of a water insoluble constituent, in particular for separating gasoline from natural gas. It comprises treating the gas mixture with activated carbon, which is saturated with water or contains a minimum of 30 per cent. of water calculated on the dry weight of the carbon. Water is supplied to the carbon during absorption to remove any heat generated. The greater part of the absorbed material is removed by direct contact of the carbon with hot water or steam, the carbon being subsequently cooled by direct contact with cold water. The carbon thus regenerated and being substantially saturated with water, is in suitable condition for treating a further quantity of natural gas from which no gasoline has been removed. H. S. G.

See also Abstracts Nos. 635 and 673.

Motor Spirit.

634. Dew Points of Air Gasoline Mixtures from Distillation Curves. O. C. Bridgeman. *Ind. and Eng. Chem.*, **20**, 8, 821.

The Engler distillation is taken as the standard of estimation of volatility of motor fuels, but this does not absolutely indicate suitability for volatilisation in engines. When the distillation figures are corrected for loss by addition at the lower end and the unvolatilised portion left as residue in the flask, the curve becomes a more true representation of volatility at 1 atmosphere pressure. The points of real interest are the equilibrium vapour-liquid curves for from 8 : 1 to 20 : 1 air petrol mixtures.

In the paper it is shown that dewpoints obtained by the Sligh apparatus are related to the 90 per cent. A.S.T.M. distillation point. Since the dew-point is the temperature at which an infinitely small amount of liquid is in

equilibrium with the vapour phase and this state is almost impossible to detect on the Sligh apparatus, the volumes evaporated at different temperatures and air mixtures was observed and the results extrapolated to 100 per cent. evaporated.

Numerous tables of data obtained are given and the results computed with the work of Stivenson and Babor. The conclusions drawn are that the dewpoints of any gasoline commercially available can be computed in any desired air-gasoline mixture from the corrected 90 per cent. figure for the A.S.T.M. distillation. A. R. S.

635. The Vertical Steam Still as Applied to Fat Oil Distillation Service. E. G. Rogatz. *Refiner*, 1928, **68**, 6, 7.

In general the stripping phase of the fat oil distillation operation consists of the introduction of a pre-heated feed into the top of a stripping section with the subsequent removal of its gasoline content through a counter-current scrubbing action. The latter is obtained from saturated or superheated steam admitted to the bottom of the column. The feed is usually preheated to such an extent that the lightest fractions of the gasoline spontaneously vaporise at the feed plate.

It is recognised that more complete stripping can be obtained under conditions of lower column pressure, but the additional consideration that all sections of the column should be held at a maximum temperature is not so generally understood. Now the best theoretical stripping column layout would contain heating coils of adequate size on each stripping tray, but if this is impracticable, it is necessary to investigate the feasibility of using at least two sets of heating coils; one near the top of the stripping section for supplying the latent heat, and the other at the bottom of the column for maintaining a relatively high average temperature through the unit.

If the lean absorption oil leaving the column is to be stripped of substantially all of its gasoline content, then the steam leaving the column can only be partially saturated. Further, the quantity of steam required per barrel of absorption oil handled, at a given temperature Tx, will be independent of the concentration of gasoline in the original fat absorption oil. Since the stripping column is uniformly heated to a definite temperature Tx, whatever the gasoline concentration in the fat oil, the same quantity of steam should always strip the same quantity of absorption oil to substantially the same degree of leanness. The basis for this assertion lies in the fact that the natural gasoline fractions dissolved in an absorption oil obey Raoult's law. It is further shown that for a given temperature Tx the consumption of steam in a perfect column will be directly proportional to $\frac{\pi}{P}$ where π is the total pressure carried on the column and P is the absolute vapour pressure at Tx of the heaviest fraction stripped from the absorption oil. This relationship gives a good test of stripping column efficiency, for if the absolute pressure on a given system is reduced by say 20 per cent. then the quantity of steam required for effecting a given degree of stripping of any particular fat oil should also be reducible by 20 per cent.

Experience has proved that the best absorption oil is a partially volatile material which yields heavy overhead fractions from the stripping column. This requires the addition of a fractionating section to the still for the removal of these heavier fractions from the finished gasoline. This fractionating section of the still operates similarly to any other temperature controlled fractionating column, there being a substantial temperature gradient between the top and bottom trays. This constitutes one of the principal differences

between the fractionating and stripping sections of the still—for whereas in the fractionating sections this temperature gradient must be maintained in the stripping sections, the best results are obtained when all such temperature gradients are suppressed. S. W. C.

636. Refining of Hydrocarbon Oils. E. B. Phillips. U.S.P. 1,687,992, October 16, 1928.

The invention describes an improved method of removing sour constituents from petroleum distillates such as gasoline. A continuous stream of the dry oil is passed upwards through a body of finely divided cupric chloride, and the reaction products from this treatment are removed by subsequent passage of the treated oil through finely divided fuller's earth. W. H. T.

See also Abstracts Nos. 626, 628, 630, 633, 666, 676 and 677.

Kerosine.

637. Technique and Economics of the Edeleanu Process for Refining Mineral Oils. G. Cattaneo. *Zeit. für die gesamte Kälteindustrie.* Sept. 9, 1928.

In the usual chemical refining of distillates, the latter are treated with sulphuric acid when unsaturated and asphaltic substances are soluble, while paraffin and naphthenic hydrocarbons are almost unattacked. This process, however, entails considerable loss.

Dr. L. Edeleanu found that only the aromatic and unsaturated hydrocarbons (present in petroleum fractions) were soluble in water-free H_2SO_3, *i.e.*, SO_2, at a sufficiently low temperature. In his researches he started with a Rumanian kerosine distillate of aromatic content 25 per cent., in which removal of aromatic compounds by chemical refining was only successful when fuming H_2SO_4 was used, and a large portion of the distillate was lost. When the temperature was lowered to —10° C, and the distillate mixed with SO_2, two liquid layers were formed. The upper consisted of SO_2—saturated refined distillate (specific gravity 0·9—1·0) and the lower of SO_2 extract (specific gravity 1·3—1·4).

The SO_2 was then evaporated from both liquids so as to obtain the refined distillate and extract as finished products. The process was carried out in a closed apparatus so that the evaporated SO_2 could be condensed and recovered. From a Rumanian kerosine distillate of specific gravity 0·819, about 75 per cent. of refined product was obtained, of specific gravity 0·803, and 25 per cent. of extract of specific gravity 0·865. This new refining process was undertaken in 1908 by the Allgemeinnen Gesellschaft für Chemisch Industrie in Berlin and by a Dutch firm in Amsterdam.

The first plant was discontinuous, and unsatisfactory from an economic point of view. It consisted of a condenser, SO_2 cooler, mixer, vapour refiner and extract evaporator. Both coolers were filled with the distillate and liquid SO_2 respectively and cooled to —10° C. The cooled liquids were admitted into the mixer, agitated, and after settling, the extract and refined distillate were drawn off one after the other into evaporators. The SO_2 from the vapours was driven directly by means of heating coils into the SO_2 condenser, and the last traces were removed by suction. In this plant about 600 kgms. steam were required to refine 1,000 kgms. kerosine-distillate with 75 per cent. by volume SO_2.

In 1924 a continuous plant was operated in which only 350 kgms. steam were required to refine 1 ton distillate with 75 per cent. by volume of SO_2.

Electrically driven rotary pumps were used with a specially constructed box for the SO_2—containing liquid. The pumps needed about 5 k.w.h./ton distillate.

It was found that considerably greater quantities of SO_2 would be required in the refining of lubricating oil distillates than for kerosine distillates.

W. Hess discovered that one could work with a much smaller quantity of SO_2 if the distillate were pre-washed with the SO_2 extract solution from the previous operation, and afterwards treated with pure SO_2. Diagrams of Hess' plant, and of a Dutch plant working under the same principles, are given.

This "counter-current" process still required too much fresh SO_2 for lubricating oil distillates, and there was still the question of the solubility of SO_2 in the evaporators. The refined product at the exit from the mixer was saturated with SO_2 to the extent of 15—30 per cent. by weight according to the type of refined product, the temperature and the pressure, and this SO_2 had to be expelled by heat.

In Cattaneo's researches he realised that with a gradual evaporation of SO_2 a considerable increase of boiling point was caused which counter-acted the heat transition, thus it was clear that the application of evaporation in three stages was limited to the concentration of the solution, and about 50 per cent., *i.e.* equal parts of extract and SO_2 was the economic limit.

Curves showing the relation between the temperature of the extract solution and the parts by weight of SO_2 in 100 grms. solution indicated that the boiling point of an SO_2 extract solution with 88 per cent. SO_2 and 12 per cent. extract did not deviate very much from that of pure SO_2. "Three-stage" evaporation could bring about an economy of heat in the extract evaporator. By the addition of heat, a fraction of the liquid in the first stage was vapourised and the vapours formed were utilised in heating in the next stage.

A systematic diagram is given of an Edeleanu plant utilising evaporation in several stages. The apparatus in which the SO_2 high pressure vapours were liquefied consisted of a boiler-tube condenser. A portion of these vapours was utilised to preheat the SO_2 extract solution before entering the high-pressure evaporator. In the last stage of the "three-stage" evaporation a concentration of about 50 per cent. was produced under condenser pressure. Further evaporation was caused by heating by steam until about 15 per cent. SO_2 was present in the extract. The remainder was conveyed into the next evaporator by means of an SO_2 compressor, and the last traces were withdrawn to the vacuum evaporator.

The economic application of evaporation in several stages naturally depends on the relation between the costs of outlay and steam. It is generally found that with 50 per cent. by volume of SO_2, the application of three-stage evaporation is not recommended, with 75 per cent. by volume of SO_2 the introduction of a high pressure stage is sufficient, and with 100 per cent. by volume of SO_2 and with greater concentrations, two stages, *i.e.*, "triple effect" evaporation is necessary.

A special gas-cooler has been introduced into the plant to prevent the evaporation of low-boiling hydrocarbons during the separation of SO_2. The SO_2 must also be constantly dried to remove moisture. The various plants described in this report are elaborated by diagrams. W. S. C.

638. Improvements in Apparatus Applicable for use in Extinguishing Burning Oil in Tanks. Asiatic Petroleum Co., Ltd., and Allom Hodgson. E.P. 296,740, June 7, 1927.

A fire extinguishing apparatus comprises one or more conduits constructed of metal and covered with a fire resisting substance, connected to a chemical

foam supply, and located down the inside of an oil tank or reservoir. The lower ends of such pipes are open or perforated, thus permitting the liquid level inside the tank to be maintained in the pipes. A float is provided inside the pipe to prevent the foam from coming into direct contact with the surface of the oil, and to assist in maintaining pressure on the incoming foam. Located at convenient heights down the face of the pipes are outlets provided with doors which are normally held closed either by reason of their own weight or by a seal or plug of easily fusible material. In the case of a fire, the seals above the oil level will be melted and the doors will open and allow the foam to flow smoothly and freely through the outlets and over the surface of the burning oil. H. S. G.

639. REFINING OIL. H. F. Gloir and O. E. Bransky. U.S.P. 1,683,289, Sept. 4, 1928.

A method is described of removing wax from lighter wax-bearing, substantially uncracked oil distillates. It consists in admixing with such distillates a paraffin wax-bearing oil of higher viscosity than the burning oils, and of lower viscosity than the oil under treatment, chilling to 0° or —10° F. and then filter-pressing the mixture. H. S. G.

640. A COMPOUND OF KEROSINE AND ALCOHOL. W. Huessy. E.P. 267,530, March 10, 1926.

Kerosine and commercial alcohol having a water content of 5—50 per cent. are mixed together and heated to 160° C. under a pressure of 10 atmospheres. The vapours are passed through a catalysator containing metallic lead heated to 400—500° C., and the vapours condensed. A motor fuel is prepared by adding to the condensate 20—50 per cent. by volume of petrol or benzol. H. S. G.

641. REFINING OILS. W. K. Lewis. U.S.P. 1,686,493, Oct. 2, 1928.

Oil to be purified is passed continuously through successive units of a cycle in each of which it is agitated with a change of filtering clay or other absorbent material, by means of a mechanical stirrer or inert gas introduced through a perforated pipe, then settled and decanted. As the clay becomes inefficient each unit in turn is by-passed and the clay therein washed with naphtha and dried by hot gases before being removed and replaced by fresh material, each unit being restored back in the treating cycle in turn. H. S. G.

See also Abstracts Nos. 619 and 621.

Fuel Oil.

642. PREPARATION OF FUEL OIL. W. S. Hughes and J. Harrop. U.S.P. 1,686,491. Oct. 2, 1928.

In the preparation of a substantially ash free fuel oil, the residue from the distillation of a hydrocarbon oil in the presence of an alkali passes from the last still in a bench through a cooler to a mixer where it is fluxed with 5—50 per cent. of gas oil or other convenient material. It then flows to a second mixer where it is emulsified with an approximately equal quantity of water, and from there passes to a third mixer where sufficient dilute sulphuric acid is added to break the emulsion. After standing in a separator the aqueous layer is withdrawn and the oily layer may be redistilled for the recovery of the fluxing oil and organic acids leaving a purified residue. H. S. G.

See also Abstracts Nos. 675 and 681.

Lubricating Oil and Wax.

643. A New Process for the Purification of Oils. Pierre Pingault. *La Revue Petrolifere*, No. 290, Oct. 13, 1928.

The majority of methods available for the purification of used oils depend on either centrifugal or filtration processes. Centrifugal processes can only be satisfactorily operated at all times on stationary plant and must frequently be preceded by chemical treatment. In ordinary filtration processes the size of the pores of the filter must be similar to or less than the dimensions of the particles to be removed, and as the filter becomes choked with impurities, the throughput rapidly diminishes.

The new procedure, which is due to M. Audibert, takes advantage of the well-known fact that in suspensions, colloidal solutions and emulsions, a difference of potential exists between the disperse and continuous phases. If the surface of a filtering medium, which has been charged positively, for example, by a suitable chemical treatment, is brought in contact with a liquid containing negatively charged particles, the particles under the influence of the electric field form a felting in which the particles have preserved their negative charge at the surface of the filtering partition provided that the radii of the capillary canals of the filtering partition are not greater than the limit of action of the electrostatic field. If the filtration pressure is not too great, the filter will not choke and the particles will not weld together in an impermeable mass under the pressure. Moreover, the first layer of impurities exercises a repulsion on the particles still remaining in the liquid, so that the latter do not adhere to the former and are arrested at a minute distance from the filter. If the particles do tend to weld themselves together in a compact layer, it is then advantageous to give the filter a charge of the same sign as that of the particles. This procedure enables not only the solid particles to be held back without choking the filter, but also permits the removal of the small liquid spheres of an emulsion. The latter is of considerable importance in the filtration of transformer oils.

A calculation is given showing that a pressure of 17.5 grams per sq. cm. can be applied to the filter without overcoming the electrostatic repulsion produced by a charge equivalent to a single layer of ions at the interstices. It would appear that this small pressure would cause the filter to have a very small throughput, but with this type the capillaries of the filtering medium can have a diameter ten times greater than that of the particles to be removed; whereas in an ordinary filter the diameter of the capillaries should be not larger than that of the particles.

The filter described consists of a pack of rectangular pieces of suitably treated fabric traversed by three slotted metal tubes, held at their extremities by two flanges, which also serve to hold the pack together. Between each rectangle of fabric a small cross piece of metal some tenths of a millimetre in thickness is placed. This is so arranged that filtration takes place upwards in the direction of the weave of the fabric. The impurities to be removed are deposited on the lower surface of the filter in such a condition that they can readily be removed by scraping. Diagrams outlining the method of construction are given.

Particular reference is made to the use of this so-called electrocapillary filter for the purification of used automobile engine oils and results showing its efficiency are tabulated.

R. W. L. C.

644. Lubrication Round-Table Discussion. *Ind. and Eng. Chem.*, **20**, 843.

St. Louis Meeting of the American Chemical Society.

Viscosities of Lubricating Oils at Low Temperatures and the Cranking Effort Necessary to Rotate cold Automobile Engines.

D. P. Barnard described some experimental work carried out by the Standard Oil Company of Indiana for the purpose of determining the viscosities of lubricating oils at temperatures down to 0° F. (17° C.), and also the effect of viscosity and pour point upon the resistance to cranking offered by the oil.

The results indicate that the cranking effort depends almost entirely on the viscosity of the oil apart from its cold test, while the circulation of the oil depends largely on its temperature being above its pour point. Parsons and Taylor first published results obtained by comparing oils from asphalt and paraffin base crudes in a Saybolt viscometer at 0° F. or —17° C. under different pressure heads.

The asphalt-base oils apparently conformed to Poiseuille's law in that the flow varied directly as the pressure drop. In the case of the paraffin-base oils the flow increased more rapidly than the pressure drop, indicating that the oil at this temperature was a plastic material, the apparent viscosity of which decreased with increasing shearing stress. A similar method was followed in the experiments now described and the observed rates of flow were plotted against the pressure drop on logarithmic scales. On such a graph any liquid which follows the laws of viscous flow will give a straight line with a slope of unity. None of the oils examined showed such a slope at low temperatures. The results are also plotted on a chart which was originally developed independently by Herschel and MacCoull, on which the temperature-viscosity graph is ordinarily a straight line. The apparent viscosities, Saybolt, of a number of oils at low temperatures, under pressure heads of 0·7, 1·4, and 27·7 kilograms per sq. cm., are given together with the value obtained by extrapolation from the results at higher temperatures plotted on the Herschel chart.

The experiments which were made to determine the effect of oil viscosity on cranking effort were carried out on engines situated in a cold room. The engines were rotated by an electric cradle dynamometer through a gear-box situated outside the cold room. The data obtained indicate consistently that the cranking efforts were determined by the oil viscosities corresponding to fairly high shearing stresses and that within the range studied the pour point of the oils appeared to have no effect on the ease of starting. R. W. L. C.

645. Cutting Oils—Their Characteristics and Manufacture. G. W. Cupit, Jr. *The Refiner*, 1928, **65**, 6, 7.

Cutting oils when mixed with warm or cold water in any proportion, readily form stable, rich, milky emulsions. Sulphur is a very important constituent of these oils, and may be present in the combined or the free state. The following method for estimating free sulphur was found to give reliable results. About 25 gr. of the oil and 100 c.c. of a solution of sodium sulphite containing 15 gr. Na_2SO_3 are placed in a 300 c.c. Erlenmeyer flask and heated under a reflux condenser for 24 hours. The flask and contents are occasionally shaken, especially during the first hours of the operation, and after digestion the aqueous solution is drawn off and 100 c.c. distilled water added to the oil. The flask and contents are then again heated for 2 hours on the steam bath. The oil and water are separated, the former is dried, and analysed for sulphur, as well as a sample of the original oil. The difference between the results is the free sulphur content of the oil. The aqueous solution is cooled and filtered. To 10 c.c. of the clear filtrate are gradually added 5 c.c. conc. sulphuric acid, with shaking. If upon standing, a more or less heavy precipi-

tate of sulphur is formed, free sulphur is contained in the oil, and the extracted oil should be analysed as directed above. If only a faint turbidity is noticed, no free sulphur is contained in the oil, and the analysis of the digested oil may be omitted.

The procedure for making a soluble cutting oil is given as follows :—

1. Mix 1 vol. of soap solution with 4 vols. of the mineral oil.

2. Add Elain to the mixture until the product is clear, and determine the amount of Elain added.

3. Test the solubility of a small sample of the mix. If insoluble in water, the concentration of soap solution should be lowered by adding dilute alcohol to the mix and clearing the produced turbid mix by gradual addition of Elain. If soluble to a rich milky liquid proceed as follows :

4. Make a " base " by mixing the soap with Elain in the proportions found under 2 (or 3, if diluted with alcohol).

5. Mix one volume of this " base " with 3 volumes of the mineral oil. A clear mix should result or the amount of Elain used is too low and should be corrected.

6. Prepare an emulsion from 5 c.c. of soluble Cutting Oil from (5) and 95 c.c. of water. Let stand. If an upper layer appears or if the emulsion gives a permanent foam upon shaking, add gradually a saturated solution of Na_2CO_3. Test by making up emulsions adding Na_2CO_3 solution until the emulsions are permanent.

The sensitiveness of an emulsion of soluble cutting oils to added electrolytes depends upon the amount of electrolyte added and upon its character. Low temperature seems to favour the action of electrolytes.

A good quality cutting oil can be made from Texas Gulf Coast Crude. This is charged to the still and distilled. The first cut overhead is gas oil, and is taken off until the distillate reaches 60 vis. at 100° F. Saybolt at the worm end. Cut No. 2 is cut into the 250 stock tank at 60 vis. at 100° F. Saybolt and out at 115 vis. at 100° F. at the worm end. Cut No. 3 (250 vis. distillate) 115 vis. at 100° F. Saybolt to 22.5° Bé on the worm, and cut No. 4 (heavy 250 stock) 22·5° Bé to 19·5° Bé on the worm are also made. Cuts 2 and 4 are combined and called 250 stock, and should be 130—135 vis. at 100° F. Saybolt. This oil contains from 0·4 to 0·6 per cent sulphur. The correct amount of flowers of sulphur is now added to the oil according to specifications. When the sulphur has been added steam is turned into the closed coils in the agitator and the temperature brought up slowly to 100° F. without agitation. Then the mass is gently agitated for about 24 hours with air. A representative sample is taken from the agitator while blowing and tested for free sulphur content. If the oil has not taken up sufficient sulphur to meet the specification range, 1·0 to 1·24 per cent., agitation should be continued without allowing the temperature to rise more than 15—20° F. When the sulphur content is correct, the mass should be smooth, homogeneous, possess a dark amber colour and be of 135—145 vis. at 100° F. In order to remove the undesirable odour of the sulphur approx. 4 oz. of Oil of Sassafras per barrel of cutting oil made should be used and added prior to shutting off air agitation. Pine tar has often been used instead of Oil of Sassafras, and where this is the case the high saponification value present may be due to either the pine tar odouriser or the oxidised mineral oil present. S. W. C.

646. Research on the Application of Used Transformer and Switch Oils. Tyko. *Brann. Chemie.* 1928, **9**, 346–348.

Previous methods of the application of used transformer and switch oils are discussed shortly in this article and new methods are divided into two groups, *viz.*, chemical and physical tests.

1. Physical Tests.

The specific gravities of transformer and switch oils always increase with use. The oils have to be freed from water before they can be further utilized.

The viscosities of such oils increase greatly in practice especially if soaps are formed. According to Schendell the permissible limit is 11 degrees Engler at 20° C.

There is no change of flash point of used transformer and switch oils. The velocity of flow of the oil drawn into the transformer or apparatus must not be less than 80 kV/cm. This stipulation was made by the Verbandes Deutscher Electrotechnik (VDE). Figures below this value show that the oil needs drying or mechanical refining.

2. Chemical Tests.

Of these the acid number is the most important and should not exceed 1·0. The saponification value is difficult to determine.

The miscibility of the used oils with various oils has been tested, but sludges have usually resulted. W. S. C.

647. Measurements and Observations on Discharge-Stability Tests on Technical Insulating Oils. W. Zimmermann. *Petr. Zeit.*, 1928, **24**, 1213–1230.

About 140 tests have been carried out on commercial insulating (transformer and switch) oils, using the S.S.W. apparatus. This differs from previously described apparatus in that a fixed potential difference is employed together with a variable electrode gap. A 110–220/30,000 v.-transformer is used, in the low-tension circuit of which a circuit breaker is included. This ensures an immediate cessation of the discharge following each individual test, thus affording protection both for the electrodes and for the oil. A prolonged discharge causes a diminution in the breakdown potential gradient ("discharge-stability") of the oil under test. A low figure is also obtained if the oil, previous to the discharge, is submitted for a prolonged period to a potential difference. Not only do different samples of oil from the same consignment show widely different discharge-stabilities (thus necessitating working with a truly representative sample and not a "snap" sample), but an individual portion of oil, when repeatedly submitted to the test without being removed from the apparatus, shows great variations. The minimum value is usually within the first few figures obtained, often the first result is the lowest, but even the tenth may be the minimum. When the first test gives the lowest figure this is not necessarily due to an incomplete cleansing of the electrodes. It is doubtful whether the first figure obtained should be neglected. Since the minimum value may fall as low as 50 per cent. below the mean of a series of determinations it is questionable whether such a mean value is an adequate criterion for an oil. Exchange of moisture between the atmosphere and a sample of oil has a great influence on discharge-stability. In a long series (100–150) of discharges there is a tendency for the discharge-stability to rise, with the usual variations in individual values, until the mean figure reaches the maximum obtainable by intensive drying. The steepness of the curve of average values gives an indication of the original moisture content of the oil or of its ease of desiccation. Used oils may be brought up to their original insulating quality by treatment with floridin or with floridin and acid. Benzine or commercial spindle oil has the same discharge-stability as the usual run of insulating oils, whilst petroleum is twice as stable. W. S. N.

648. Improvements Relating to the Removal of Paraffins from Fluid Hydrocarbons. Akt. Separator-Nobel and K. G. Malm. E.P. 296,805, June 7, 1927.

A method of separating paraffins from liquid hydrocarbons is described, according to which the paraffins are first precipitated at least partly in the crystalline state by slow chilling. They are then separated from the liquid hydrocarbons (to which one or more diluents have been added) by means of a separator provided with mechanical means for carrying away the separated paraffins. A suitably constructed centrifuge is that described in E.P. 10,874/05. H. S. G.

649. Improvements Relating to Centrifuging Wax. C. Boesch. E.P. 287,094, March 13, 1928.

This is a method of purifying wax in which the wax is first heated in a waterbath and then placed in the drum of a centrifuge. The latter is of the type wherein turbine blades are rigidly attached to the bottom of a perforated drum, rotatably supported within a casing, so that the steam supplied to the turbine rotates the drum. The exhaust steam heats the materials contained therein, and is subsequently discharged together with the separated substance and the condensation water, through a common outlet in the casing. By centrifugal force the mixture of liquid wax and water is forced through a fine mesh sieve and perforated drum. Wax and water from the discharge are collected in a vessel where the wax accumulates on the surface of the water. H. S. G.

650. Removing Amorphous Wax and Asphaltic Material from Oil. T. Clarkson and H. B. Heal. U.S.P. 1,686,437, Oct. 2, 1928.

A lubricating oil fraction is treated with 60-80% of a light petroleum distillate, and refrigerated to substantially below the freezing point of water to cause solidification of the greater portion of the amorphous wax and asphaltic material. It is then subjected to pressure filtration through fine filter paper or filtering medium until a substantial quantity of solidified material has accumulated. Filtration is then discontinued and a hot light petroleum distillate is passed through the filtering chamber, but not through the filtering medium, in order to dissolve the solid matter. H. S. G.

651. Means for Preventing Sludging in Transformer and like Oils Maintained at High Temperatures. The British Thomson-Houston Co., Ltd., and D. C. Cox. E.P. 278,365, Sept. 28, 1927.

The useful life of an insulating or other oil which is kept heated for long periods of time is greatly extended by providing a charge of fuller's earth in contact with the oil. A transformer tank containing transformer elements surrounded by a quantity of insulating oil contains at the bottom, a suitable container holding a body of fuller's earth having a volume of about 6 per cent. of that of the oil. H. S. G.

652. Apparatus for Purifying Used Lubricating Oil. E. L. Gross. U.S.P. 1,681,980, Aug. 28, 1928.

This invention consists of a device for purifying used lubricating oil, being particularly applicable for the purification of oil used for lubricating internal combustion engines. The apparatus may also be installed under the hood of an automobile, where it will, while the engine is running, intermittently draw the used oil from the bottom of the crank case, free it from gasoline, water and all suspended matter and finally return it in a purified condition to the upper part of the crank case through an oil filter. H. E. T.

653. Oil Reclaimer. A. L. Henny. U.S.P. 1,684,270, Sept. 11, 1928.

An oil purification apparatus of the distillation type, susceptible of being applied to the reclamation of used crank case oils is described. In reclaiming used lubricating oil the diluents such as gasoline are first removed, leaving the residual oil in a condition for further treatment, such as the removal of solid impurities. The device is capable of being used in conjunction with internal combustion engines in order to overcome dilution of the lubricant. H. E. T.

654. Montan Wax. A. Riebeck'sche Montanwerke. A.G., E.P. 288,258, Mar. 12, 1928.

The mixture of steam and solvent vapour produced in distilling the extracted wax and by steaming the extracted coal is passed through a dust separator to an insulated collecting main. The mixed vapours then pass to the brown-coal drying apparatus, where their latent heat is used in drying the coal. The hot condensate collects in a separator, the solvent then returning to the extraction apparatus. When a water-soluble solvent is used, the effluent from the drier or separator is led to a column still without previous cooling. G. S.

655. Process and Apparatus for Reconditioning Mineral Oils. C. J. Rodman and Mas. Hecht. U.S.P. 1,685,681, Sept. 25, 1928.

A process and apparatus are described for reconditioning mineral oils, in particular electric insulating and transformer oils. The process is carried out as follows:—

The oil to be reconditioned is preheated and passed through a system of parallel heaters where it is kept in a state of agitation. The agitation of the oil is increased by the combined ebullition, degasification and vapourisation of the contained impurities. Under the conditions in which the oil is heated, moisture mechanically held in the oil, dissolved gases such as air, oxygen, nitrogen, etc., are continually eliminated and are removed by a vacuum pump, thus leaving the oil in a thoroughly evacuated condition. The evacuated oil is now treated with an absorbing material out of contact with air; the oil and absorbing material being thoroughly mixed in a tank by means of a centrifugal pump. The oil is now passed from this tank through a series of filters where the absorbing material is removed, leaving the used oil in thoroughly reconditioned state. H. E. T.

656. Improvements in and Relating to Lubricating Oils. T. H. Sanders. E.P. 297,201, Sept. 20, 1928.

The improvement consists in adding to the lubricating oil an organic ester of low viscosity, such as amyl acetate or butyl lactate, the quantity added exceeding 1 per cent. It is claimed that by this means the gumminess of the oil is reduced and that the starting of internal combustion engines in cold weather is greatly facilitated. H. E. T.

657. Process of Treating Slack Wax. B. L. Souther and W. A. Gruse. U.S.P. 1,685,058, Sept. 18, 1928.

Slack wax is comminuted to form a fluid magma of sufficient fluidity to be handled like a liquid at ordinary temperatures. The bulk of the oil present can then be removed by mechanical treatment. The separated wax is treated with an amount of a suitable solvent, preferably an alcohol having from 3—6 carbon atoms to the molecule, sufficient to dissolve the oil but not the wax. Separation is then effected by any suitable means. H. S. G.

658. Treating Slack Wax. B. L. Souther and W. A. Gruse, Assrs. to Gulf Refining Co., Pittsburg. U.S.P. 1,685,008, Sept. 18, 1928.

In a process for the manufacture of wax, a petroleum distillate is chilled to crystallize the wax which is pressed to separate oil from a solid slack wax containing oil. The slack wax is comminuted until it is converted into a fluid consistency capable of being pumped, and the solids and liquids are mechanically separated by means of a filter press or centrifuge. H. S. G.

659. Improvements in Lubricants. Standard Oil Development Co. E.P. 297,068, May 9, 1927.

The film-maintaining property of a lubricating oil is considerably increased by adding to a hydrocarbon oil (*e.g.*, of 285 Saybolt viscosity at 100° F.) an amount of an alkali soap less than ½% by weight of the oil, and heating the mixture to a temperature above 350° F. but not above the boiling-point of the oil. The soap may be added in a finely divided condition, or may be formed in the body of the oil by adding thereto the desired base and fatty acid or fatty oil in substantially combining proportions. Alternatively, the soap may be introduced in aqueous solution (conveniently 10%) or as a concentrated solution or gel in oil to the desired amount and mixed with the lubricating oil in absence of air. H. S. G.

660. Treating Petroleum Products. E. R. Wiles. U.S.P. 1,684,426, Sept. 18, 1928.

A method is described of recovering amorphous paraffin of high melting-point from petroleum. It comprises treating the petrolatum with a mixed solvent consisting of approximately 35 per cent. acetone and 65 per cent. benzol. The solution is then cooled, and the undissolved paraffin separated therefrom, the solvent from the separated paraffin and from the solution being recovered by evaporation. H. S. G.

See also Abstracts Nos. 639, 641, 664, 665, 668, 689 and 670.

Special Products.

661. A New Method for the Production of Greater Stability of hot drawn Tar-, Bitumen- and Asphalt-Pitches. G. Kropfhammer. *Erdöl und Teer.*, 1928, **29**, 513.

The stability of hot pitch is very important in the building of roads. The combination of hot drawn pitch with the road surface is also of primary importance since the latter must be cleansed free from dust before applying the hot layer. This is difficult to carry out, for although the principal quantity of dust can be removed, finely powdered particles of dust still remain and set solid in the surface.

If it is desired to remove this very fine dust, skilful washing with water is found to be impracticable, as the ease of wetting even the finest sand is extremely small. This small-wetting capacity is more marked in the case of road-paving with hot tar, bitumen or asphalt. It is clear that the more intimate and solid the union of the tar, bitumen, or asphalt with the road surface the less is the formation of waves or holes on the surface.

The wave-motion in tars, bitumens and asphalt-pitches is chiefly due to the continuous rolling load of traffic on its surface. The waves occurring in this way are impressed by the traffic particularly in the cold season, and cause cracks in the surface as the pitch has not such a satisfactory ductility as in the warm season.

These disadvantages are overcome by applying an intermediate layer which is treated with a wetting medium before adding the hot pitch. These "wetting media" are well-known liquids possessing strongly emulsifying qualities and which adsorb in the layer even colloidal dust particles. Hexaline or methylhexaline meet these requirements. One of the substances is added to the hot pitch and a bitumen or tar-emulsion is thus formed. In this way a complete wetting of the stone surface is ensured, and the bitumen from the emulsion enables a further cementing of the hot tar, bitumen or asphalt pitch with the road-surface. W. S. C.

662. Utilisation of the Products of Azneft's Asphalt-Beton works. *Oil Bulletin*, 1928, No. 19, p. 12.

Baku commune is paving their roads with asphalt—a product of the Asphalt-Beton works. The work is carried out exclusively by mechanical means and is 50 to 60 per cent. dearer than the ordinary Baku macadam, but nevertheless makes it possible to have a far more durable road. The Baku-Balachani chaussé, a distance of 13 klm., is now being asphalted. T. W.

663. Manufacture of Asphaltic, Bituminous or Tarry Emulsions. Société Anonyme La Trinidad. E.P. 279,026. *Applic.* (*U.K.*), June 13, 1927.

Emulsions intended for the construction and maintenance of roadways consist of Trinidad asphalt mixed with pulverent or colloidal mineral substances and judiciously fluxed with a residual petroleum oil to bring it into a suitable condition. To this mixture, maintained at a temperature of from 100°-150°C., is added 3-12 per cent. by weight of an anhydrous soda or potash soap to which has been added from 1-8 per cent. of its weight of commercial olein. To the homogeneous mixture the desired concentration of water may be added which can reach up to 300 per cent. by weight to obtain an emulsion. H. S. G.

See also Abstracts Nos. 640 and 645.

Analysis and Testing.

664. The Dependence of Temperature on Viscosity. I. C. Walther, *Erdöl und Teer.*, 1928, **29**, 510-511.

The previously known formulæ connecting the viscosities of mineral oils with temperature are discussed, especially Vogel's formula $\eta t = \eta D \frac{t-t_1}{t-tD}$ where t_1 is the temperature at which viscosity η in absolute units is equal to that of water of 20·2°, therefore = 1.

tD the temperature at which η is infinitely great.

ηD the viscosity at tD.

This gives no information on the knowledge of viscosity, but only a mathematical conception of the viscosity-temperature curve.

Walther derives a formula by measuring the viscosities at two temperatures (50° and 20°, or 50° and 100°) by which a single constant is determined, indicating the variation of viscosity with temperature.

The temperature-viscosity (in absolute units) curve should be a hyperbola, and the logarithm of the logarithm of the viscosity as abscissæ plotted against the temperatures as ordinates should give a straight line.

The following formula is proposed for mineral oils:—

$$\log.\ \log.\ V_K = \frac{-M(t+50)}{100} + \log.\ \log.\ V_K\ 50°$$

where V_K represents the kinematic viscosity.

M represents the logarithmic temperature factor of the viscosity. M is calculated by subtracting the log. of the log. of the viscosities at two temperatures, dividing this by the temperature difference and multiplying by 100. There is only one constant (M), except at 50°. Walther found a value of 0·569 for M.

He applied his formula to several oils and promises the results in a further report. W. S. C.

665. The Dependence of Temperature on Viscosity. II. C. Walther. *Erdöl und Teer.*, 1928, **30**, 526-28.

In Walther's first paper (*cf. Erdöl und Teer.*, 1928, **29**, 510-511) the formula $\log\ \log\ V_K = \frac{-M(t-50)}{100} + \log\ \log\ V_K\ 50°$ was derived in which V_K is the kinematic viscosity, M the logarithmic temperature factor of the viscosity, *i.e.*, the decrease of log log V_K/100° C. The present article deals with the application of this expression to various types of mineral oils.

1. American Oils.

Frank and Selberg applied the above equation to a series of American mineral oils. The specific gravities and viscosities at 50°, the values of log log V_K 50° and M are given in Table 1. The oils are arranged in order of increasing viscosities and M is calculated from the viscosities at 20° and 80°.

Table 1.

No.	9. Amer. spindle oil.	11. Amer. machine oil.	6. Amer. machine oil.	10. Amer. machine oil.	4. Amer. refined machine oil.	12. Amer. refined machine oil.
	2/50	4/50	6–7/50	9/50	20/50	15·5/50
Specific gravity 20° ..	0·9804	0·9174	0·9272	0·9636	0·9427	0·9211
Viscosity E 50° ..	2·08	4·18	7·33	10·21	25·54	14·49
log log V_K 50° ..	0·041	0·173	0·241	0·274	0·359	0·309
Temp. factor M ..	0·580	0·580	0·576	0·584	0·579	0·520

It is interesting to note from the table that M for all six oils, excluding number 12, M has the same value, 0·580 $\pm$ 0·004.

The graphical relation between log log V_K (as abscissæ) and temperatures, 20–80° (as ordinates), produces parallel straight lines.

2. Russian Oils.

The results with Russian oils are given in Table 2.

Table 2.

No.	2 Russian spindle oil.	1 Russian machine oil.	3 Russian machine oil.	7 Russian cylinder oil.	8 Russian motor oil.
		4·1	6·5	10–11·5	16/17
Specific gravity 20° ..	0·8959	0·9008	0·9048	0·9104	0·9091
Viscosity E 50°	3·05	4·23	6·65	13·17	17·06
log log V_K 50°	0·125	0·174	0·230	0·301	0·325
Temp. factor M	0·588	0·567	0·544	0·525	0·516

The above Russian oils, unlike American oils, show a decrease in M with increasing viscosity. When log log Vκ is plotted against temperature, the viscosity curves represent straight lines which are not parallel but approach one another with decrease in temperature. On the other hand, when the values for M are plotted against the values of log log Vκ 50° the points lie on a straight line. Thus M = a log log Vκ 50° + b where a and b are constants.

3. Viscosity and Specific Gravity.

For Russian and American oils the specific gravities increase in the same order as the viscosities. The graphical relation between log log Vκ and specific gravities represents almost straight lines.

4. Specific Gravity and Temperature Factor.

When the values for M as abscissæ are plotted against specific gravities as ordinates, the points lie as expected on a straight line.

It is clear that the "typically Russian" and "typically American" mineral oils obey definite laws, but number 12 of the American oils occupies quite a separate position.

In conclusion the results serve to show the value of an oil as a lubricant. Since that oil is the most suitable as a lubricant whose viscosity changes least with temperature, it follows from the previous considerations that of two oils with equal viscosities at a given temperature, that oil is more suitable for lubrication purposes whose temperature factor is less.

Tables 1 and 2 show that the Russian oils in question with the higher viscosities are better for lubrication than the corresponding American mineral oils, since these oils with the higher viscosities give less values for M than the American oils. W. S. C.

666. The Application of the Valenta Reaction to Gasoline-Benzol Mixtures. Weller. Autotechnik, Feb. 1928, p. 7.

The Valenta dimethyl-sulphate test depends on the differing solvent power of dimethyl-sulphate for aromatic and saturated open chain hydrocarbons. The aromatics are completely soluble at room temperature, while the paraffins are supposed to be practically insoluble. The author, however, has shown that both the soluble and the insoluble fractions of a gasoline were practically identical with the original spirit. With definite mixtures as, for instance, 50 per cent. standard gasoline and 50 per cent. benzol, the dimethyl-sulphate extract contained fairly large amounts of gasoline along with the benzol, while the undissolved portion still contained benzol even after shaking out a second time. The author concludes that the dimethyl sulphate number is useless for judging a motor gasoline. W. H. T.

667. Flash-point Determinations in the Normal Flash-point Apparatus. G. Meyerheim and F. Frank. *Petr. Zeit.*, 1928, **24**, 1235-1237.

Flash-points have been determined of a number of insulating and heavy lubricating oils using the following flash-point apparatus: (1) Marcusson's apparatus with crucible of (*a*) porcelain, (*b*) Krupp's V2A steel, (*c*) brass; (2) the Schlüter-Göckel apparatus, which has a clearance of 2 mm. between the thermometer bulb and the side and bottom of the crucible; (3) an apparatus made by Sommer and Runge to Bochow's design, with similar clearance of 2 mm. or 5 mm., this clearance being accurately reproducible by means of a collar and flange fixed on the thermometer, and a socket in the crucible lid. Good agreement was obtained using these different apparatuses. W. S. N.

668. A Precision Pipette Viscometer. S. W. Ferris. *Ind. and Eng. Chem.*, Sept. 20, 974.

A pipette of 5 ccs. capacity is drawn out gradually at the point to an inside diameter of 2 mms., after which it is polished flat through a plane at right angles to the axis of the pipette. The upper end of the pipette is fitted with a fine mesh copper gauze to prevent access of grit and the whole immersed in a water jacket fitted with stirring gear. The oil to be tested is held at the desired temperature for 10 minutes before the determination and the time of efflux between marks at the top and bottom of the pipette measured.

It has been found that with any oil sample or samples the time of efflux is plotted against Saybolt seconds, the points all lie on a straight line which intersects the Saybolt axis.

It is claimed that conversions from pipette seconds to Saybolt may be made with any mineral oil or compounded oil by use of a straight line calibration curve, that the determinations are quicker and more accurately repeatable and that the pipette is no more fragile than the Saybolt instrument.

A. R. S.

669. Determination of Unsaturateds in Lubricating Oils. F. S. Bacon. *Ind. and Eng. Chem.*, Sept. 20, 971.

These results and method are published that they may aid the systematic study of high molecular weight olefines and the value of unsaturated bodies in lubricating oils.

25 ccs. of 10 per cent. sulphuric acid are placed in a separating funnel with 1 to 10 gms. of the oil, which is diluted with 10 ccs. of pure benzine. Small quantities of $\frac{1}{2}$ normal bromide-bromate solution are added, with vigorous shaking after each addition until a slight excess of free bromine is shown by the colour of the oil. Potassium iodide is now added and the solution back titrated to excess with thiosulphate. This excess is necessary so that the thiosulphate may destroy any iodine dissolved in the oil layer. The water layer is now drawn off, washed and back titrated with iodine to an end point with starch.

If B is the bromine number per 100 gms. of oil, M the mass of sample, TN, T_1N_1, T_2N_2 the titrations and normalities of bromate-bromide, thiosulphate and iodine respectively, then

$$B = \frac{8{\cdot}0}{M}(TN + T_2N_2 - T_1N_1).$$

To determine the percentage unsaturateds calculated on the mean molecular weight of sample, Menzie's method of observing the change of vapour pressure on addition of the substance to a boiling solvent was adopted, as it was found to give good checks, although the method is open to criticism.

The percentage of unsaturateds was calculated from the following formula.

$$\% = \frac{\text{Br. No. x mol. wt.}}{160}$$

Tables of data obtained are given.

A. R. S.

See also Abstracts Nos. 634 and 647.

Refinery Plant.

670. Refrigeration for Oil Refining. N. H. Hiller, jr. Paper read before the Fifteenth Western Meeting of the American Society of Refrigerating Engineers, *Cold Storage and Produce Review*, **31**, Oct. 18, 1928.

Reduced paraffin-crude distillate is re-run in fire stills to convert the amorphous wax to a crystalline form more amenable to filtration. The

34°–27° Bé fraction is purified in agitators and piped to the chilling plant which may be one of two types.

The "double pipe chilling machine" consists of an 8 in. pipe surrounding a 6 in. pipe, cooling medium being circulated between the two pipes. A screw conveyor, driven by chain sprockets on the extension of the shaft at one end, is situated inside the 6 in. pipe to move the congealed oil along with the wax. Usually up to 12 chiller sections are connected in series, a standard machine containing approximately 50–55 sq. ft. of surface. The rate of heat transfer between oil and cooling medium of a double pipe chilling machine is around 4–5 B.Th.U. per sq. ft. per ° F. of mean temperature difference per hour. At this rate one section will chill approximately 25 barrels of distillate from 100° F. to 20° F. per 24 hours. Each machine must be carefully insulated, and should be provided with a large excess of power to overcome the heaviness and hardness of the wax which crystallizes out.

The second type of chilling machine is similar to an ice cream freezer, consisting of two vertical shells, in the inner of which is a power driven paddle wheel arranged so that upon being revolved, the inertia of the oil presses the blades against the shell and thus keeps it free from a deposit of wax. Cooling medium is circulated between the two shells. This type of machine is very convenient for use down to 20° F.

It is usually necessary to remove the wax in two or three stages, and may be carried out by cooling the paraffin distillate from 90/100° F. storage temperature to 55° F. for the first chilling, pressing under 500 lbs. sq. in. and returning the wax free distillate to the chilling plant where the temperature is brought down to 20° F. After pressing the wax free distillate may be further chilled if necessary.

Heat interchange plays an important part in the economics of the process. The ideal chilling plant contains two sets of refrigerating machinery to take care of the varying loads at the different temperatures. The design of the plant should enable minimum storage necessary between each pressing operation. Steam ram pumps maintain the necessary circulation.

H. S. G.

671. Oil Storage Tanks. I. R. Afonin. E.P. 298,262, Aug. 6, 1927.

The evaporation of oil in storage tanks is prevented by covering the surface of the oil with separate but flexibly connected floating cells. The downward inclined sides of each cell are adapted to slide in contact with the inclined sides of adjacent cells. The cells are preferably in the form of parallel-pipedons, except those in contact with the wall of the tank. If it is desired that the sides of the peripheral cells incline in the same direction to the wall of the tank, each row of cells should contain one cell of trapezoidal form, two opposite sides being inclined in opposite directions. Containers for water may be provided on the cells and means may be also provided to prevent water penetrating between the cells. G. S.

672. Method and Apparatus for Distilling Petroleum Oils. J. E. Bell. U.S.P. 1,683,151, Sept. 4, 1928.

This invention relates to improvements in the fractional distillation of petroleum oils and distillates. The still is provided with a tower through which a helical passage passes, the feed being introduced at or near the top of the tower, and passing down over the turns of the helix, while the vapours from the still pass upwards in the reverse direction, the heavier reflux and unvaporised portion of the feed entering the still from the tower, while the lighter vapours from the still and feed escape from the top of the tower as the desired fraction. In this manner effective heat interchange and refluxing of

the oil is brought about, making close fractionation of the oil possible as well as a greatly increased rate of distillation. In practice the distillation can be so regulated that the temperature of the reflux entering the still approximates to the temperature of the oil in the still, so that almost all of the overhead distillate from the tower is distilled in the tower itself from the fresh feed introduced. The vapours may be passed at high velocity so as to sweep the liquid surfaces with which they come in contact, and promote heat transfer between the liquid and vapours with a minimum friction loss and a maximum surface of liquid subjected to the action of the vapours moving with high velocity. By operating the tower in such a manner that preheated oil is introduced at an intermediate point, while a lighter oil or distillate is introduced at the top of the tower, the capacity of the tower can be greatly increased, while more effective fractionation can be obtained.

The towers used in the present invention may be used in a battery of stills when exceptionally close cuts can be obtained. Various modifications of the tower used in this invention are described and diagrams are given of each. H. E. T.

673. Means for Separating the Oil from Oil-laden Gas. H. A. Fraser, W. L. Price and J. Hetherington. E.P. 297,622. Sept. 27, 1928.

A process is described for separating oil from oil-laden gas, and is primarily designed for use in connection with refrigerating plants in which a gas is compressed by a pump, liquefied by a condenser, and regasified in an evaporator, the process being a continuous cycle of operations in which the gas in its passage through the pump carries with it some of the lubricating oil.

To effect the separation of the oil from the oil-laden gas, the gas is passed through a chamber in the gas passage, packed with tubular pieces of metal upon which the oil is deposited. The deposited oil flows into a chamber having an outlet automatically controlled. H. E. T.

See also Abstracts Nos. 635, 638, 649, 674, 678, 679, 682 and 685.

Engineering.

674. Heat Transfer in Oils Flowing through Pipes. By M. Garcia. *Ind. & Eng. Chem.*, Sept., 20, 1928, 889.

The writer shows that the basis of the design of heat transfer equipment, the logarithmic mean temperature difference equation, is based on assumptions which, while satisfactory for water and gases, are unjustified when dealing with oil and other viscous liquids.

For petroleum oils variations of the specific heat—temperature relation and the viscosity temperature relation are of such an order that the assumption of constancy is entirely unjustified.

Differential equations have been derived taking into account the change in physical properties of the liquid with the temperature.

The writer emphasises that the variations of the viscosity-temperature relation is of much greater import than that of the specific heat-temperature relation.

Various examples are given showing the errors incurred by the use of the logarithmic mean temperature difference equation. Graphical methods are given for the calculation of mean liquid temperatures and mean temperature differences for use in the design of heat exchangers for viscous oils over large temperature ranges, or in the correlation of experimental data. W. L.

675. EXPERIMENTS ON A SOLID INJECTION ENGINE. By F. Mucklow. *Automobile Engineer*, Aug. 1928.

The engine used for the experiments was a single cylinder Crossley solid-injection engine, type 0123, bore 14 ins., stroke 23 ins., compression ratio 10·3 : 1, the normal speed 212 r.p.m., maker's rating 66 b.h.p.

The rocker arm operating the fuel pump was mounted on an eccentric spindle so that the pump timing could be varied over a range of some five degrees crank angle while the engine was running.

Four series of trials were run, each series being at a different load. In each series the fuel injection timing was varied over a range of approximately ten degrees crank angle; the effect of these variations on fuel consumption, efficiency, and heat losses were observed, the effect on the process of combustion was followed by the Hopkinson optical indicator.

As the time of injection is advanced from 6° to 17° before top dead centre the lag between injection and ignition shows a slight increase, especially at high loads. The rate of pressure rise, following the start of combustion and the indicated maximum pressure increases, uniformly as the injection is advanced.

With advance in injection the brake thermal efficiency increases, while the fuel consumption is reduced, the effect of the first five or six degrees advanced is considerably greater than that of the remainder of the range investigated.

The heat losses to the walls and breech remain constant over the range investigated, while the percentage loss to exhaust rises as the injection is retarded; the temperature of the exhaust gases showed little change over the range.

In general it would appear that the best results were obtained with the injection timing fined by the makers, *i.e.*, 348·5°. W. N. H.

See also Abstracts Nos. 661, 670, 671, 687 and 690.

Anti-Detonation.

676. IMPORTANCE OF MIXTURE RATIO IN RATING FUELS FOR KNOCK. John M. Campbell, Wheeler G. Lovell and T. A. Boyd. *Ind. Eng. Chem.*, 1928, **20**, 10.

The authors describe a series of tests made on a variable compression engine with a bouncing pin indicator, to show that mixture strength variations may lead to discordant results between various laboratories, even though the experimental technique is otherwise good.

The fuels under examination were tested at a compression ratio adjusted to give a fairly loud knock, and bouncing pin measurements were taken at a series of fuel feeds for each.

Mixture ratios were determined by exhaust gas analyses.

Comparisons were made between ethyl fluid-standard spirit blends and benzine-standard spirit blends, and also between ethyl fluid blends and *n*-heptane-iso octane blends.

The results show that true comparisons can only be made if the mixture strength of each blend is adjusted to give maximum knock for that blend, and that if either constant air-fuel ratio or maximum power is adopted as the condition at which knock is measured, the sensitivity and accuracy of test suffers. R. S.

677. Effect of Volatility of Petroleum Fractions on Detonation Value. J. C. Geniesse and H. F. Huf. *Ind. and Eng. Chem.*, 1928, **20**, 8.

Five petroleum naphthas were each cut into six fractions by simple fractionation, close fractionation being avoided.

The A.S.T.M. distillation data of the original samples, the fractions, and some blends are tabulated.

Each fraction and blend was tested in a single cylinder engine by the throttling method at a constant air-fuel ratio of 12·5 to 1, the detonation values being expressed as per cent. increase or decrease of average absolute pressure in the intake manifold for audible detonation compared with the value for a standard reference fuel.

It is concluded that there is a definite relationship between the detonation value and

(*a*) The specific gravity of a fuel;

(*b*) The 50 per cent. distillation temperature.

A graph is constructed from the experimental data from which the effect of changing the volatility of spirit from a given source may be predicted.

R. S.

See also Abstract No. 626.

Cracking.

678. Process and Apparatus for Cracking Oil. G. Egloff. U.S.P. 1,682,742, Jan. 13, 1928.

In a cracking plant consisting essentially of cracking tubes, expansion chamber and dephlegmator, the vapours in the upper part of the expansion chamber are mechanically agitated by fans or other rotating elements.

H. G. S.

679. Apparatus for Cracking Oil. G. Egloff and W. R. Howard. U.S.P. 1,683,766, 1928.

The deposition of carbon in cracking tubes is prevented by the insertion of a helical agitator which is caused to rotate by the passage of oil through the tube.

H. G. S.

680. Apparatus for Cracking Hydrocarbons. E. C. Herthel. U.S.P. 1,683,184, 1928. Appl., July 11, 1927.

The apparatus comprises a pressure still containing a bed of filtering, absorbent material, *e.g.*, fuller's earth, extending across the still and below the normal liquid level therein, heating tubes, dephlegmator, pumps and condenser. The charging stock, *e.g.*, gas-oil, is introduced into the top of the dephlegmator and is pumped through the heating coils in admixture with oil taken from the portion of the still below the filtering bed. From the coils, the oil re-enters the still above the bed of fuller's earth, the overhead being taken to a condenser and the run-back, after filtration, returned to the heating coils.

H. G. S.

681. Process for Treating Residual Oils. L. C. Huff. U.S.P. 1,683,826, 1928. Appl., May 26, 1924.

The residual oil from a cracking operation is introduced before any substantial cooling has occurred, into an enlarged zone in which a pressure materially lower than that prevailing in the cracking plant is maintained and

in which distillation occurs. The unvaporised portion is withdrawn from the base of the enlarged zone and is used as refinery fuel, the excess being returned to the zone in such a way that the body of oil therein is maintained in a turbulent condition. H. G. S.

682. Improvements in the Production of Liquid and Gaseous Hydrocarbons of Low Boiling Point from Mineral and other Oils, Tars and the like. I. G. Farbenindustrie Akt. E.P. 295,974, April 21, 1927.

Hydrocarbons of high boiling point may be converted into hydrocarbons of low boiling point by means of one or more heating devices immersed in the liquid under treatment. The heating elements are exposed to a continuous or intermittent blast of gases which contain oxygen or hydrogen or of other gases or vapours such as steam or ammonia which are free from carbon and are capable of giving off oxygen, the blast being heated preferably to about 500° C. The heating device is preferably of small dimensions compared with the amount of material to be decomposed, and may consist of a short length of pipe maintained in a state of incandescence by internal heating, or of a rod, ribbon or spiral of metal or other conductive material such as silicon carbide, graphite or charcoal made red or white hot by electric heating. H. S. G.

683. Improvements in the Method of Working with Hydrocarbons at High Temperatures. I. G. Farbenindustrie Akt. Original specification, E.P. 296,683, March 3, 1927 (1). Subsequently divided into E.P. 296,693 (2), E.P. 296,694 (3), E.P. 296,752 (4), and E.P. 296,753 (5).

(1) In operations conducted with gaseous or liquid hydrocarbons at elevated temperatures, other than the conversion of hydrocarbons of high boiling point into others of lower boiling point, and carried out without an addition of considerable amounts of hydrogen, it is important that the gases or vapours, so long as they are at an elevated temperature, should only come into contact with such materials as do not produce a deposit of coke or soot. Iron, nickel and cobalt in particular should be absolutely excluded from the hot parts of the apparatus which are most suitably constructed of the noble metals, ferro-silicon, and alloys of chromium, manganese, molybdenum and cobalt, or steels containing chromium, manganese or tungsten. The use of a catalyst may not be necessary, but if present should similarly not be such as to give rise to coke or soot formation, but should consist of the materials mentioned above, or silica, pumice, barium carbonate, charcoal impregnated with barium salts, boron, tungsten or the like, or coated with lustrous carbon.

(2) The inner surfaces of apparatus coming into contact with liquid or gaseous hydrocarbons at elevated temperatures consist of aluminium in order to avoid coke or soot deposition.

(3) Any surfaces which come in contact with liquid or gaseous hydrocarbons at high temperatures, whether metallic or non-metallic, are provided with a coating of lustrous carbon of the type described in the "Berichte der Deutschen Chemischen Gesellschaft, vol. 56, p. 2071, and vol. 59, p. 2433.

(4) In processes involving working with liquid or gaseous hydrocarbons at an elevated temperature, coke or soot deposition is avoided by adding or supplying one or more metalloids, namely, phosphorus, boron, arsenic, antimony, bismuth, phosphorus, selenium or a compound thereof, or silica, to the metallic surfaces of the apparatus or parts thereof which come into contact with the liquid or gaseous hydrocarbons at an elevated temperature,

mere traces of such additions often being sufficient. Such additions may be effected by alloying materials such as iron or copper with boron or the like, or coating such materials with a thin layer of the substance by wetting with a solution of boric acid or other soluble compounds of the metalloids, or treating them with gases containing boron or arsenic or the gaseous compounds of the metalloids, or by adding to the hydrocarbons to be treated small quantities of volatile compounds of the metalloids. In this latter case, it may be advantageous to carry out the process in the presence of hydrogen.

(5) In order to avoid the deposition of coke or soot when working with liquid or gaseous hydrocarbons at elevated temperatures all metallic parts coming into contact therewith should be constructed of molybdenum, tantalum or chromium. H. S. G.

684. Improvements in the Manufacture and Production of Low Boiling-point Oils by Cracking Mineral Oils, Tars and the Like. I. G. Farbenindustrie Akt. E.P. 272,483. Appl. May 26, 1927.

The formation of coke deposits in cracking processes is eliminated, or the carbon is deposited in colloidal form, by effecting the heating of the liquid materials, with or without the presence of hydrogen or other gases or vapours under any suitable pressure, by means of electrically heated bodies, kept in motion and entirely submerged in the liquids, and having a sufficiently high temperature to effect cracking. Preferably, use is made of a stirrer the vanes of which are electrically heated and having the simplest possible shape to facilitate the provision of a scraper which renders the deposition of carbon incrustations impossible. The heating bodies and inner surface of the heating vessels should be constructed of materials not capable of becoming charged with electricity of opposite polarity to that of the carbon suspended in the liquids. Such metals which do not form sulphides, oxides or carbides under the conditions of working are suitable for the purpose. Separated carbon may be removed from the oil by any suitable means; for example, by means of filters, preferably such as have an electrically opposite character to that of the carbon in the oil, or by means of electrophoresis. H. S. G.

685. Apparatus for Treating Oil. R. T. Pollock. U.S.P. 1,683,801, Sept. 11, 1928.

An apparatus is described for cracking oil. It comprises a furnace containing a heating coil the discharge side of which is connected to an expansion chamber provided with a residue draw-off line and vapour outlet. The latter is connected to the side of a dephlegmator near the lower end. The dephlegmator consists of an elongated drum with partition members near its upper and lower ends respectively, connected by a series of small vertical tubes surrounded by a helical baffle.

The upper part of the intermediate chamber is provided with a vapour outlet pipe connected to a condenser coil. The whole system is adapted to work under a pressure of 150 lb. Raw oil is fed through the tubes in the dephlegmator and thence to the main feed line and inlet side of the heating coil. Oil in the liquid phase is discharged into the vaporisation chamber from which vapours pass into the central chamber of the dephlegmator where the heavier fractions condense whilst at the same time preheating the new oil. H. S. G.

See also Abstract No. 693.

Oil Field Technology.

686. Water Control Problems Analysed. R. Van A. Mills. *Oil and Gas J.*, **27** (18), 38.

The paper deals generally with a preliminary survey of water encroachment and methods of control. The problem is divided into two divisions: (1) without artificially increasing the pressure in the sands, (2) by artificially increasing the pressure. Each division is subdivided according to conditions existing in various fields.

The effective pressures occurring in the sands are briefly surveyed.

Edgewater encroachment in sands under gas and hydrostatic pressures is investigated and the relationship between these pressures necessary for minimum encroachment is discussed.

The effect of the viscosity of oil under gas propulsion is considered as is the rate of fluid movements in the sand. The writer concludes that gas pressure is the most effective medium for water control, and the best available propulsive agent in recovering oil from the sands. W. L.

687. Oil Well Cements. F. G. Tickell and P. O. Wankowski. *Oil Field Eng.* Sept. 1, 1928.

Hydraulic cement is generally agreed to be the most suitable material to support the casing of the well bore and prevent the movement between one stratum and another. The properties most desirable in the cement are slow initial set, early hardening and strength, density, fluidity when slurrified, and impermeability.

The intital set is said to be accelerated in the drilling due either to water being squeezed from the slurry—a very improbable happening—or due to the pressure exerted interfering with the chemical reactions involved.

Experiments were carried out to throw light on the problems of initial set. The first was a measurement of the differences of electrical resistance as the slurry hardened, but no inflections on the curve corresponding to the initial setting as determined by the Vicat needle could be noted.

Secondly, it is claimed that the heat generated by cement in setting, due either to exothermic reaction or heat of solution of the crystalline parts of the slurry, begins at the time of initial set. Experiments by both the writers and Butler have shown that this statement is not verified by practice.

The effect of pressure on initial set was determined by using the Vicat apparatus electrically controlled inside an autoclave, so that the electric circuit in which there was a galvanometer was broken from the time the needle touched the cement surface till it had travelled 35 mms. The initial set was said to have occurred when this time was 30 seconds. With pressures up to 1,500 lbs. the pressure was found to have little effect on the initial setting time.

The results of temperature on time of set are given below, as determined by Bachman, and it is concluded that this effect has more to do with initial set than any other.

Water %.	% Calcium Chloride.	Hours after gauging to attain final set.				
		70°F.	90°F.	110°F.	130°F.	150°F.
45	0	8.95	8.30	8.03	7.03	5.47
45	3.25	4.72	3.97	3.13	2.88	3.00

By immersing the standard moulds for breaking stresses in water for three days under various pressures and determining the tensile strength immediately after removal, it was found that maximum strength was reached under

1,000 lbs. pressure. The falling off in strength of the samples treated above this pressure may be due to disintegration on removal from the pressure autoclave. A. R. S.

688. Emba. Prospecting in the Temir District. *The Oil Bulletin.* 1928, 20, 13.

Embaneft proposes to commence prospecting work in June, 1929, in the Temir district, with a view to supplying the Tashkent and Turkestan-Siberian railways with oil fuel.

Work will first be undertaken in the Djaman-Agatch area with two well drilling rigs. After completing the prospecting at Djaman-Agatch the work will be transferred to Ita-Sai. It is proposed to acquire the Doherty Stone Drilling Co.'s rigs as being most suitable for prospect drilling. The total preliminary expenditure is estimated at 0.5 mill. rbls. T. W.

689. Recovery of Petroleum from Oil-bearing Sands. H. Atkinson. E.P. 296,213. Oct. 10, 1927.

Oil remaining in oil-bearing sands after substantial exhaustion is recovered by introducing into such sands an alkaline solution of sufficient strength to displace the oil. If the oil sands are flooded with water, a saturated solution of caustic alkali or soda ash is added in sufficient quantity to diffuse through the body of water and oil remaining in the sands and liberate the oil. The solution is introduced into a well which communicates with the oil-bearing sands and oil displaced is extracted from an adjacent well. A sufficient hydrostatic head to effect diffusion is maintained by filling the well with such solution to a suitable height and maintaining a constant level. An aqueous soap solution or solution of a suitable salt capable of modifying the surface tension at the oil-water interface may also be used. H. S. G.

690. Tubing Catchers for Oil Wells. S. V. Dillon. E.P. 298,320. Aug. 9, 1927.

The tubing catcher includes a slidable wedge block and a slip assembly which is supported in the well casing by friction means. The wedge block rests on the slip assembly and forces it down the casing as the tubing descends. The wedge block functions to force the slips into locking engagement with the casing. The tubing may be moved through the catcher to a limited extent before and after it is anchored. The catcher will operate while the tubing is in position in the well, or when it is being lowered or raised. G. S.

See also Abstracts Nos. 631 and 691.

Petroleum Geology.

691. Measurement of Terrestrial Radioactivity. *Oil Field Engineering.* Vol. 4, No. 3, 21.

This article is a commentary on Information Circular 6072, published by U.S. Bureau of Mines. The circular consists of translations of papers by Russian authors on measurements of terrestrial radioactivity and a paper by F. W. Lee on radioactive substances and the methods for locating them.

The possibilities of the use of radioactivity in prospecting for petroleum deposits are mentioned, experiments having shown that penetrating radiations increase over oil strata due to the high absorbing power of oil and also to

the layers underlying the oil being richer in radium owing to their formation from colloidal sea ooze.

The method of radiometric contouring is briefly described.

Measurements show that at any particular station, the intensity of penetrative radiations is constant, but that the intensity may change sharply, in quality and quantity, within a few metres. Experiments seem to point, to these fluctuations being due to the deeper strata only.

Mr. Lee's paper contains details of photographic, ionization and calorimetric methods of measuring radioactive substances. W. L.

See also Abstract No. 627.

Coal and Shale.

692. Oils from Shale, Lignite and Coal. J. W. Horne and A. D. Bauer. *Oil & Gas J.*, 1928, pp. 168-177.

The paper gives a detailed account of an investigation carried out jointly by the United States Bureau of Mines and the State of Colorado on the yields and properties of oils produced from shale, lignite and sub-bituminous coal, using the standard assay method of the Bureau. The samples tested were obtained from the States of the Rocky Mountain region and comprised 10 of oil-shale, 14 of lignite, 16 of sub-bituminous coal and 2 of bituminous coal. The sources and analyses of the raw materials are given in detail, together with the yields and properties of the products but the report cannot be embodied in an abstract. H. G. S.

693. Treatment of Shale. W. H. Hampton. U.S.P. 1,687,763, Oct. 16th, 1928.

Shale-like bituminous solids are digested in a finely-divided condition with a heavy mineral oil at 500° to 700° F. Steam is mixed with the vapours and the mixture cracked at higher temperatures and the products fractionally condensed. The residual digestion mixture is diluted with a lighter oil, the solid matter removed, and a heavy fraction recovered from the residual oil. This heavy fraction, together with a heavy fraction from the cracking products, is used for digesting a further portion of the raw material.

The digestion mixture may be cooled, the solid matter separated, the liquid material being employed in cooling the digestion mixture, thereby heating the liquid. The liquid is then fractionated at a suitable temperature. G. S.

See also Abstract No. 654.

WORLD POWER CONFERENCE.

Fuel Conference, London, 1928.

The following abstracts of the more important papers dealing with the various branches of the petroleum industry, and which were read before the Fuel Conference, have been prepared by W. H. Thomas, A.R.S.M.

694. The Economics of the Oil Industry. E. H. Davenport.

This paper deals with the fluctuations of the gasoline market and their causes. The major problem of the oil industry since the war has undoubtedly been that of producing sufficient fuel for the ever-growing automobile industry. The enormous increase in demand has been so successfully met that the price of gasoline shows the least increase since 1913 of any commodity, in spite of the increase in cost of drilling and refining. This result has been achieved, firstly, by increasing the production of crude oil; secondly, by increasing the yield of gasoline by cracking; and, thirdly, by the development of the natural-gas-gasoline industry. Thus, the increased efficiency of the production and refining processes has greatly cheapened the price of gasoline. The extent to which these methods have assisted is shown by the facts that since 1916 the production of natural-gas-gasoline has increased by 1·473 per cent., and that of cracked gasoline by 1·359 per cent.

Fluctuations in the price of gasoline have broadly followed those of the raw material, but the chance discovery of prolific sands in the Seminole area can be quoted as an example of a case where competitive drilling broke the market in crude oil and reduced prices by more than half. Drilling in the United States has become more expensive each year, due largely to the greater drilling depths to which wells are drilled. The average cost of drilling a well is given as $18,725 in 1927 as against $4,765 in 1913. Of the cost of producing a barrel of crude oil in America, only 22½ per cent. is due to direct oil-raising expenses, and only 13·2 per cent. due to dry holes and abandoned wells. The average barrel of crude oil may be taken to yield 27 per cent. of gasoline, 10 per cent. of kerosine, 46 per cent. of fuel and gas oils and 7 per cent. of other products. If this average composition be considered, and if the costs of manufacturing these products be taken at those proportions of the total price realised, represented by their proportionate yields from the crude oil, it will be found that some of the products would be selling at a dead loss. Gasoline as a rule must carry a price which will return a much greater proportion of the total receipts than is represented by its proportionate production from the crude oil. The author is of the opinion that the supply of gasoline from other sources is not likely to influence the price of petroleum gasoline for a considerable time.

695. The Oil Industry in Japan. Keizaburo Hashimoto.

A brief resume of the oil industry in Japan is given pointing out the distribution and geology of the oil-fields, the producing methods in use and the refining methods employed. The adoption of new methods and plant has been responsible for the development of the natural gas and cracking industries.

Japan, faced by a steady decrease in crude oil production, erected her first natural gasoline plant in 1914, and her first cracking plant as early as 1912. By this means the yield of gasoline from the crude oil had been more than doubled in six years.

696. The Production of Gaseous Fuel from Liquid Fuels. J. Kewley.

It is stated that considerable quantities of gas are now being made from oil and distributed from central gas works in localities where oil is cheaper than coal. Oil gas plants are of three types: those which produce enriching gases, those which make oil gas instead of coal gas in central works, and those which make the gas on the site where it is to be used. The oil gas may be produced either by direct cracking or by burning a part of the gas to supply the heat necessary to crack the rest or finally by making gas by the interaction of highly-heated oil with superheated steam. The author states that the consumption of oil on large plants is 5·65 imperial gallons per 1000 cu. ft. of gas (500-600 B.Th.U.s per cu. ft.). In the direct cracking process, working is intermittent, the chambers being heated by air blast containing a little oil. Continuous plants are built which work on the principle of effecting cracking by the heat supplied by partial combustion of the oil. Reference is made to the Hakol-Zwichy process in which conditions are controlled so that there is no deposition of carbon. When the gas is used for direct firing, about 95 per cent. of the heat value of the original oil is available in the form of hot gas, while, if cooled, about 80 per cent. is available. The Dayton plant which operates on the same principle is very compact, while the Goldsborough process specialises in the production of high calorific value oil gas.

697. Chemical Composition of Polish Natural Gases and the Determination of Their Calorific Value. K. Kling, L. Suchowiak, W. Lesniasnki, K. Katz, W. Dominik and J. Wójcicki.

Part 1: Natural gas is abundant throughout the entire area of the Polish oil fields situated in the Carpathian lowlands. As it is used practically solely as fuel for the production of power for the oil industry, analyses of the various combustible constituents present are of considerable interest. Part 2: The only methods available for the determination of the calorific value of natural gas, such as calorimetric and analytical methods, do not readily lend themselves to quick determination at the actual source of supply. By means of the so-called "densoscope" it is, however, comparatively easy to ascertain the relative density of the issuing gas, and as natural gas consists almost entirely of paraffin hydrocarbons, the calorific power can be calculated from the density observed, provided that the gas is not in any way adulterated. Part 3: The determination of calorific power is also rendered difficult by the smallness of the sample available. In such instances a method is given for calculating the calorific value from the chemical composition.

698. The Composition and Chemical Properties of Russian Oil. J. Elin.

An exhaustive study of the composition of petroleum offers great difficulties on account of its extreme complexity and the inactive nature of its components. The petroleum from the principal Russian oil fields is generally classified as naphthenic, but this is only partly correct, for, although naphthenes predominate in the lightest fractions of the Baku oil, paraffin hydrocarbons are also present. The principal Russian oil fields give roughly 95 to 97 per cent. of the total Russian production, and the crude oil from these fields can, with few exceptions, be classified as mixed base oils. All contain wax, some in very small quantities, and most have a considerable percentage of gummy

and asphaltic substances, low acidity, small sulphur content and low percentage of unsaturated hydrocarbons as well as wax. It is thus possible to prepare lubricating oils ranging from middle oils to bright cylinder stock from the residuum. They are obtained by distillation, using a slight vacuum and introducing large quantities of super-heated steam. The distillates require little treatment.

699. The Characteristics of Motor Spirit. Gesellschaft für Wärmewirtschaft.

A specification on specific gravity alone has been found to give little indication of quality and the peculiarities which manifest themselves during consumption. Numerous experiments carried out in conjunction with producers and users of motor spirit have led to various useful conclusions and suggestions. Although the fractional distillation curves give a truer indication of the characteristics, it is necessary at the same time to quote specific gravity, as variation from heavy to light grades give rise to trouble with the setting of the carburetter float. The temperature at which 95 per cent. boils over will be considered as the "final boiling point," and as all fractions with a boiling point of 100° C. or less show no appreciable difference in character, they will be quoted collectively. To comply with the various requirements of the trade, three methods of characterisation have been decided upon, details of which will be found in the original.

700. Evaluation of Fuels used for Aviation in Germany; E. Rackwitz and A. von Philippovich.

This communication deals with the properties required in Germany for an aviation fuel for use in modern engines, and discusses the relation of these properties to engine results obtained in practice. In Germany petroleum spirit is used mainly, but lignite spirit and benzol-gasoline are frequently utilised, while the use of anti-knock dope is mentioned. The compression of German aero-engines lies between 5·5 : 1 and 6·3 : 1, and thus suitable fuel is difficult to obtain. When mixtures of benzol and gasoline are used, the former must be limited to 60 per cent owing to risk of freezing, and this limits the compression ratio of the gasoline itself to 5·5 : 1. The difficulty of obtaining a universal standard fuel for direct compression ratio tests is the expense; otherwise *n*-heptane and iso-octane mixtures would be suitable. It is realised that no definite conclusions as to the anti-knock value of a fuel can be obtained from a knowledge of chemical composition. Boiling ranges of gasoline and benzol mixtures are given, and the possibility of gassing with volatile fuels is pointed out. The relation between boiling range and compression ratio is indicated, and a rough relation between the latter and chemical composition is illustrated by a graph. The relation between spontaneous ignition temperature and composition is shown, and the effect of anti-knock dopes on ignition temperature is referred to. The risk of corrosive properties is realised and a suitable corrosion test is described. For sulphur content, a maximum figure of 0·3 per cent. is given, and the influence of latent heat when dealing with alcohol or alcohol mixtures is mentioned.

701. Properties Required of Liquid Fuels for use in Automobile and Aircraft Engines. W. Ostwald.

A convenient method of expressing the value of a fuel for use in internal-combustion engines is by means of the "energy volume"; that is, the volume of fuel required to produce a definite amount of energy. The essential features of a fuel for automotive engines are low cost and high ton mileage,

and to secure the former thermal efficiency is often sacrificed. Fuel development and engine design have influenced each other. For example, the constant-volume cycle engine has been improved by increasing the compression ratio, this being rendered possible by the production of fuels stable to high compression, such as aromatic, naphthenic or alcoholic fuels. On the other hand, the advent of the high-speed Diesel engine has resulted in lower compression ratios being used, and also in a considerable decrease in engine weight. For this type of engine the aliphatic fuels are preferred on account of their low spontaneous ignition temperatures or pro-knocking tendencies. Benzol being plentiful in Germany, automobile engines there tend to have comparatively high compression ratios. In U.S.A. the benzol production is small, so that compression ratios can only increase as better fuels become available. The three properties of a fuel which determine its value are: (*a*) volatility, (*b*) purity, and (*c*) combustion characteristics. Other properties to be standardised in addition to the above are: compression stability, stability on storage, freedom from corrosive action, sulphur content, H/C ratio, odour and stability to low temperatures.

702. The Use of Ethyl-Alcohol as a Motor Fuel. E. Hubendick.

The existence of an extensive sulphite pulp industry in Sweden makes the alcohol fuel question one of great importance for that country. It is shown that 2 per cent. of the weight of timber consumed can be obtained as pure sugar. The author believes that by improved methods a much larger yield of sugar will be obtained and, if necessary, the whole of the liquid fuel requirements of Sweden could be met by the consumption of only a small proportion of that country's output of timber. Research has produced a new denaturant in croton aldehyde, only 0·19 per cent. being required. The vapour pressure of alcohol is lower than that of gasoline, as is also the calorific value, but the latent heat is much higher. In order to obtain practical equality as regards volatility an alcohol mixture must have a higher partial pressure in the engine cylinder due to the lower air-fuel ratio that must be employed. In order to obtain a clear comparison between alcohol and gasoline, when used in an engine, the calorific value of the air fuel mixture should be determined and the consumption stated in thermal units per horse-power hour; in other words, the efficiency as well as the quantitative consumption must be considered. As long as the percentage of alcohol in a mixture does not exceed 23, with a normal engine, the engine efficiency will show no noticeable change, and full power will be obtained. With higher concentrations of alcohol, the loss of efficiency with normal carburetter setting is very great. In this case the intake air must be provided with additional heat to compensate for the high latent heat of the alcohol. The float valve must be adjusted to compensate for the greater specific gravity of the alcohol. Allowance should be made for advancing the ignition, and the size of the carburetter jet must be increased in inverse proportion to the thermal value of the alcohol fuel per unit volume. The author also deals with the question of the effect of air humidity upon the heat required for the evaporation of the fuel as it enters the engine.

703. Petrol Engines and their Fuels. H. R. Ricardo and C. Thornycroft.

This paper commences with a brief summary of the conditions controlling the power output and efficiency of any engine operating on the constant volume cycle. The authors point out that the principal controlling factor is the ratio of compression which can be employed, and that this is limited generally by the incidence of detonation. They show that detonation is

very largely a function of design and of scale effect, and that for any given fuel characteristic it is controlled by the absolute distance from the sparking-plug to the farthest point in the combustion chamber, by the nature and degree of turbulence, and by the position of the sparking-plug. It is shown that in three different engines the limit of compression for the same fuel was found to be 3·7 : 1, 5·3 : 1 and 6·8 : 1.

It is shown also that as cylinder size increases so the losses due to internal friction, direct heat loss, etc., diminish, but the tendency to detonate increases with the result that if advantage is taken of the latter feature, to employ a higher compression ratio, the efficiency is, within limits, independent of cylinder size. The influence of mixture strength on power output, ignition advance and on the tendency to detonate are illustrated by separate test figures. The influence of various fuels and groups of fuels on the tendency to detonate is discussed, and it is shown that so far as hydrocarbon fuels are concerned, mixtures obey nearly a straight line proportional law. A number of tests with metallic dopes are quoted showing that the effect varies widely with the fuel to which they are added. Finally, reference is made to the influence of fuel volatility on the performance of the engine, and it is shown that within limits the volatility of the fuel has comparatively little effect, provided that the design and heating of the induction system are appropriate.

704. The Use of Heavy Oils in Internal-Combustion Engines. E. Hubendick.

The paper deals with the use of heavy oil in internal-combustion engines of the Diesel type, and describes tests carried out on a heavy asphaltic oil with a four-cylinder, four-cycle Hesselman motor of 65 horse-power. The analysis of the fuel is first given; after which the mechanical details of construction and operation are dealt with. The method of carrying out tests is described, and the results obtained using gas oil and two types of heavy fuel oil are tabulated. It is proved by the results of the tests that a second quality oil previously used only in air injection engines can also be used for the operation of motors with direct injection, provided that the latter are constructed in a suitable manner.

705. Fuel Oil for Heavy Oil Engines. H. Moore.

The author discusses the suitability of various available oils for use in present-day internal-combustion engines and the relation of this to laboratory analyses. The requirements of a heavy fuel are intimately related to the type of engine in which it is to be used, and the special requirements of the heavy oil engine impose extra limitations on the character of the fuel. Generally, the higher the compression of the engine, the less exacting its requirements as to the chemical nature of the fuel. The user expects the fuel to be free from inorganic matter and to have a high calorific value. Again, it should burn in the cylinder with a clear exhaust and freedom from fouling the combustion chamber, so that the engine is capable of running for long periods without overhaul. The fuel should have a sufficiently low ignition temperature to be able to start it from cold, and should not congeal at ordinary low temperatures. The completeness of combustion in the cylinder can be considered only in relation to engine design, for clean combustion depends on the timing and adjustment of the fuel valve to the viscosity of the oil and to a less extent on chemical nature, particularly on the hard asphalt content. To the latter is usually attributed coke formation, smoky exhaust, and injury to exhaust valve faces, although large low-speed, two-stroke engines give better results when heavy oils of high asphalt content are used.

There appears to be no relationship between the sulphur content of an oil and its engine performance, except in so far as sulphur may be a component of hard asphalt. Apart from silencer corrosion, the presence of sulphur appears to have no deleterious effect. The spontaneous ignition temperature is the factor which differentiates between coal tar oil and petroleum fuels. The former have such high ignition temperatures that it is often found necessary to arrange for pilot ignition gear when using them, while the latter have not this disadvantage.

706. Internal-Combustion Engines with Exhaust Turbo-Supercharging. A. Büichi.

This paper deals with the application to large slow-running Diesel engines of exhaust driven superchargers of the type developed for aircraft during the war. The first part of the paper deals with theoretical considerations, and the author stresses the importance of scavenging of exhaust products, and advocates a valve timing giving an appreciable overlap between the opening of the air inlet valves and the closing of the exhaust valves. The scavenging effect obtained thereby serves both to increase the weight of air in the cylinder, and at the same time to cool the cylinder walls, piston head and exhaust valves. This compensates for the increased heat flow due to the greater volume of heat and to the higher initial air temperature. A description is given of tests carried out on a six-cylinder, four-cycle Diesel engine to which this system of supercharging was applied. It was proved that as the power is increased by supercharging, the heat flow to the cylinder walls, piston, valves, etc., is increased at a somewhat lower rate. Since the mechanical losses are virtually unaffected by the addition of the supercharger, it follows that the mechanical efficiency increases steadily with increase of load. The maximum load recorded during the tests corresponds to a brake mean pressure of about 133 lb per sq. in., which was obtained with a supercharge of 7·11 lb. per sq. in., with the turbine running at slightly over 6000 r.p.m. under these conditions.

707. The Present Position of the High-Speed Heavy Oil Automobile Engine. W. Riehm.

The paper commences with a short specification of a fuel suitable for small high-speed Diesel engines, and then proceeds to a brief description of a number of these motors developed recently in Germany, including one air injection engine, several solid injection engines, and several employing the pre-combustion chamber principle. One very interesting example is given of a high-speed, two-cycle Diesel, namely, the Junkers opposed piston type. A description is given of the various purposes to which these engines may be applied, and indicates their advantages over gasoline engines.

708. Experiments with Diesel Engine-Driven Locomotives on Russian Railways. P. Yanouschevsky.

The author deals with a number of experiments carried out in Russia with a view to the development of a full scale main line Diesel locomotive. Three types of experimental locomotive are described, the first being made up from existing units and consisting of a ten-cylinder 1000 h.p. Vickers engine and electrical drive with two generators fitted one at either end of the main ngine. No data is given as to the actual performance of this locomotive, which was built up solely for the purpose of preliminary experiment. Later, two more locomotives were built and put into service, one having electrical transmission and the other gear transmission, through the medium of magnetic clutches. Of the two, the results show a slight advantage in favour of

the geared locomotive, and from the summary it would appear that the operating cost of either is approximately 76 per cent. of that of a steam locomotive of equal capacity. The tests show a marked saving in operating cost, despite the fact that the experiments were conducted in an area where water was plentiful.

709. The Present Position of the Use of Heavy Oil for Motor Traction. M. Defays.

The author divides heavy fuel engines into three classes, viz., low-speed, moderate-speed and high-speed engines, and discusses the advantages of the high-speed type. Several moderate-speed engines are now on the market, but the Held-Rallye engine is cited as a case of the successful development of the high-speed type. If the cost of the heavy fuels of the gas-oil type remains well below that of the present price of motor fuel, the heavy oil engine will offer a distinct advantage in the matter of fuel costs. The principal economic consequences of the use of heavy oil engines for transport and the general fue of high-speed heavy fuel oil engine would be an increase in the proportion os the world supply of available liquid fuels, together with a simplification of the refining processes necessary, with a concurrent reduction in cost, and the further development in the cost of transport generally.

710. The Technical Aspects of the Storage, Handling and Transmission of Liquid Fuels by the User. I. Lubbock.

This paper gives first-hand and very useful information on the grades of fuel oil commonly used, with their physical and chemical characteristics. Storage tanks are dealt with and a typical user's installation is described, while the means of heating fuel oil, its filtration, transmission and pumping are also touched upon.

711. The Availability of the Alberta Bituminous Sands for the Production of Fuel Oil. K. A. Clark.

The purpose of this paper is to give, very briefly, a statement of the information available regarding the bituminous sands of Alberta and their potentialities as a source of liquid fuel.

712. Modern Developments in the Oil Distillation. A. E. Dunstan and S. F. Birch.

The modern aspect of distillation by means of pipe stills and fractionating "bubble" towers is described. Straight distillation of crude as well as the fractionation of the less volatile component of natural gas and of lubricating oil are dealt with. Modern pipe heaters are described in some detail, and the methods of control adopted in current practice are outlined.

713. Chemical Reactions Involved in the Refining of Petroleum S. F. Birch and W. S. Norris.

The paper deals with the reduction of sulphur content of the lighter fractions of crude oil, and gives details of the reactions which occur during the use of various reagents. The soda washing of light distillates removes hydrogen sulphide completely and mercaptans partly. The readiness with which hydrogen sulphide undergoes oxidation to free sulphur necessitates its removal at the earliest opportunity. Free sulphur reacts with sodium sulphide to give poly-sulphides which oxidise mercaptans to disulphides, the latter passing back into the oil. Sodium plumbite solution is used after soda washing to sweeten an oil which contains mercaptans, these being converted to lead mercaptides which are partly oil soluble and incompletely

precipitated. Addition of free sulphur completes the treatment, lead mercaptides being converted into insoluble lead sulphide and oil soluble disulphides. Aqueous alkaline hypochlorite solution oxidises organic sulphides to sulphones, disulphides to sulphonic acids and mercaptans first to disulphides and then to sulphonic acids. Excess of alkali stabilises the hypochlorite solution and retards the oxidation of sulphur compounds. Sulphuric acid has a general solvent action on sulphur compounds dissolved in a petroleum distillate. Mercaptans are oxidised to disulphides, which are dissolved in the acid. Sulphonation of aromatics occurs, and basic substances are removed as salts. Sulphuric acid acts as a polymerising agent for the more reactive constituents of cracked distillates, but its action is greatly dependent on the concentration and temperature at which it is used. Aqueous cuprammonium sulphate is said to remove mercaptans, disulphides, acetylenes and diolefines. Aromatic or unsaturated hydrocarbons are much more soluble than paraffins in liquid sulphur dioxide, this forming the basis of Edeleanu's process. The extract is a valuable source of anti-detonating fuel. Owing to a preferential solubility a partial desulphurisation is also effected. Anhydrous aluminium or zinc chlorides act as energetic polymerising agents for unsaturated hydrocarbons even at ordinary temperatures whilst at high temperatures they act as desulphurising agents. Roasted bauxite is a desulphurising and decolorising agent for kerosine. The action is one of adsorption but oxidation of sulphur compounds by adsorbed oxygen also plays a part. The action of silica gel is probably similar to that of bauxite. Fullers earth and floridin are used as filtration media to remove colour from kerosine, and are also employed as polymerising agents for treating cracked distillates in the vapour phase.

RECENT ADVANCES IN CRACKING. A. E. Dunstan and R. Pitkethly.

The paper commences with a general review of cracking in which it is noted that vapour phase cracking processes were the first in the field, but gave way eventually to liquid phase processes operating at lower temperatures. The Burton process was the first to produce commercial quantities of gasoline and now in addition, the Cross, Dubbs, Holmes-Manley, Jenkins and Tube and Tank are the principal processes which show continued progress on a commercial basis. Short descriptions of each of these are given, and show their similarities and differences, and indicate the type most suitable for modern conditions. Vapour phase cracking shows a much higher loss in the form of gas and the yield of spirit is less than by liquid phase cracking. The principal difference between the two methods is the temperature at which the process is carried out. In liquid phase cracking, the temperature of operation is between 380° and 480°C, whereas in vapour phase cracking, a minimum temperature of 600°C. is necessary. The long time factor characteristic of the former is absent in the latter, but no high pressures are used in the vapour phase process. Of the true vapour phase processes, three of the best known are the Gyro, the Leamon and the Knox. Short descriptions are given of these plants and methods and the paper concludes with a useful list of references.

715. THE CRACKING PROCESS. A UNIVERSAL SOURCE OF MOTOR FUELS. G. Egloff.

In the introduction of this paper, it is shown that motor fuels can be made by cracking processes from a great variety of starting materials. These include petroleum, shale oil, low temperature tars, coal tars, bituminous tars and asphalts, wood tars, vegetable oils, fish oils and oils obtained from peat, lignite, etc. It is possible to obtain by cracking, more than twice the

yield of spirit than can be obtained by straight distillation of crude oil, and this cracked spirit owing to its content of naphthenic, olefinic and aromatic hydrocarbons is especially valuable as an anti-detonating fuel. In those processes utilising residual oils, the products are gas, motor spirit, Diesel oil and fuel oil. Those working on distillate oils give spirit, Diesel oil, gas and coke. In yet another method, the products of cracking are of secondary importance, the main object being the reduction of viscosity of heavy oils. Cracking processes are nowadays sufficiently flexible in operation to allow of the production of materials in agreement with the demands of any economic conditions. Special products of high price (alcohols, solvents, etc.) can be manufactured from both the spirit and the gas from cracking operations. The author gives details of operating conditions as well as analytical data concerning the cracking of all the various types of raw material quoted above, and concludes his paper with a number of comments regarding the future of cracking. Finally a bibliography of cracking is given which completely covers the period 1926–1928.

716. Investigations in Distilling Processes of Swedish Oil Shales. B. Holmberg.

This paper gives details of the chemical examination of the fractions of the crude and the application thereof. An account is also given of the organic compounds occurring in the shale.

717. Shale and the Production of Oil from Shale. U.S. Bureau of Mines.

This contribution describes the experimental shale-oil plant established under the auspices of the U.S. Bureau of Mines in Colorado and discusses the oil-shale resources of the United States, the characteristics of American oil shale and its products. Attention is also drawn to American oil shale developments, technical and economic problems of the industry and a future programme of work.

W.H.T.

ABSTRACTS

Arranged by

W. H. THOMAS, A.R.S.M.

Abstractors :

S. F. BIRCH, Ph.D., D.I.C.
R. W. L. CLARKE, B.Sc.
W. S. CLARKE, B.Sc.
S. W. COOKE, B.Sc.
P. DOCKSEY, B.A.
W. H. FORDHAM, M.A., F.G.S.
H. S. GARLICK, B.Sc., A.I.C.
J. E. MARSHALL HALL, A.R.S.M.
W. N. HOYTE, B.A.
W. LITTLEJOHN.
W. S. NORRIS, Ph.D., D.I.C.
R. PITKETHLY, A.I.C.
G. SELL.
H. G. SHATWELL, Ph.D., M.Sc., A.I.C.
E. R. B. SKIPWORTH, B.A.
T. A. SMITH, B.Sc.
R. STANSFIELD, A.M.Inst.C.E.
A. R. STARK, B.Sc., A.I.C.
H. E. TESTER, B.Sc.
W. H. THOMAS, A.R.S.M.
T. WISKIN.

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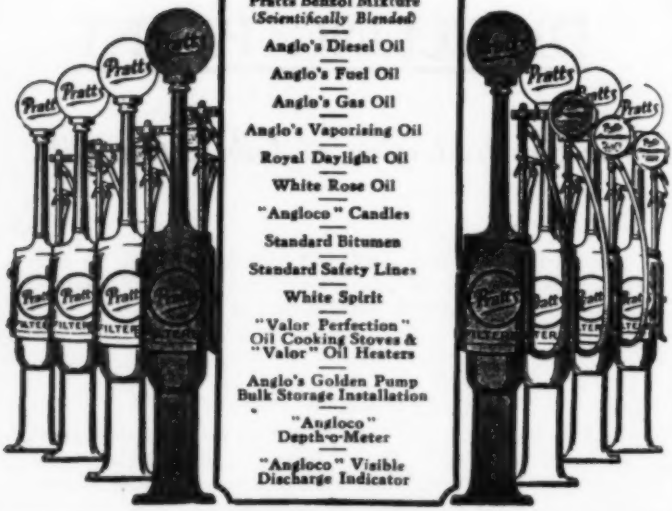
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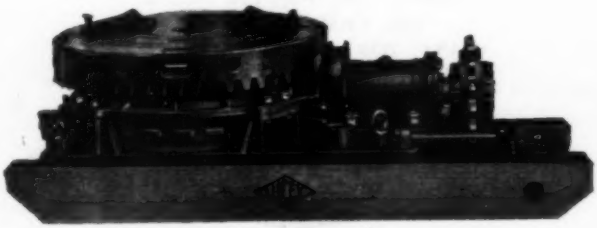

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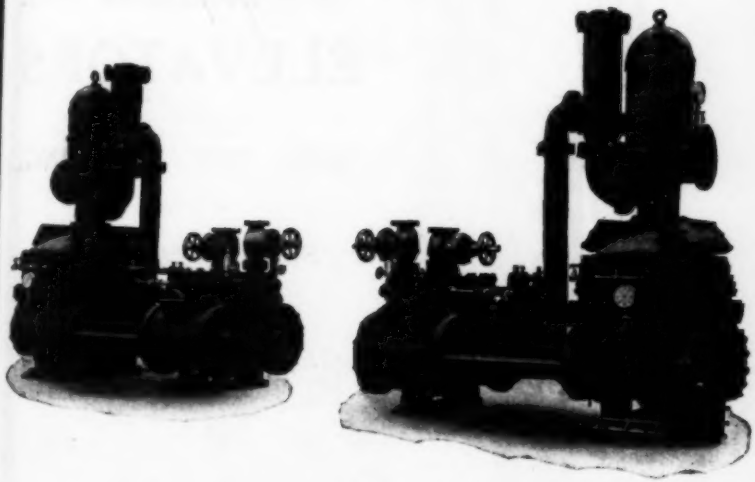

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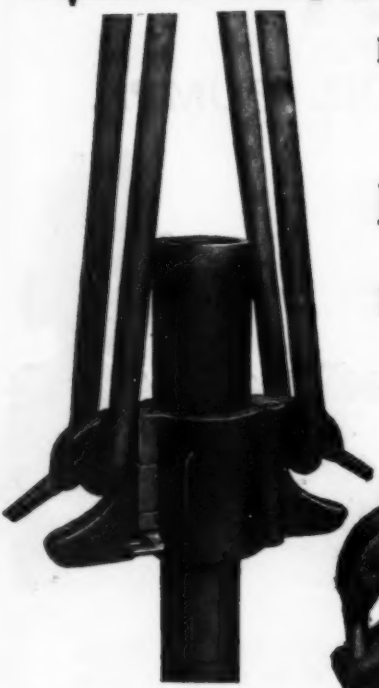

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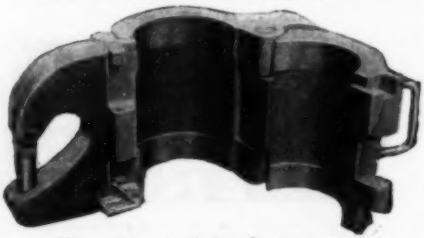

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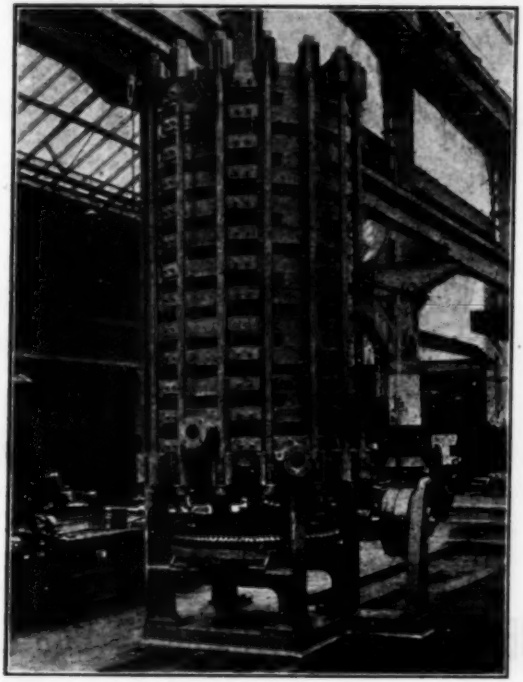

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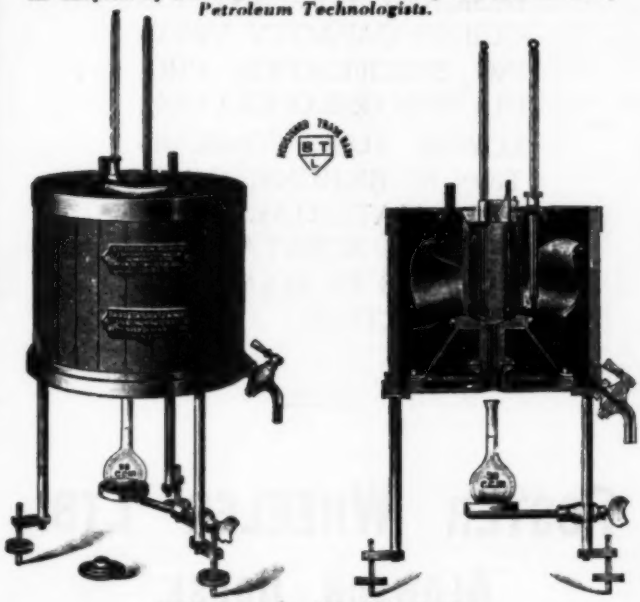

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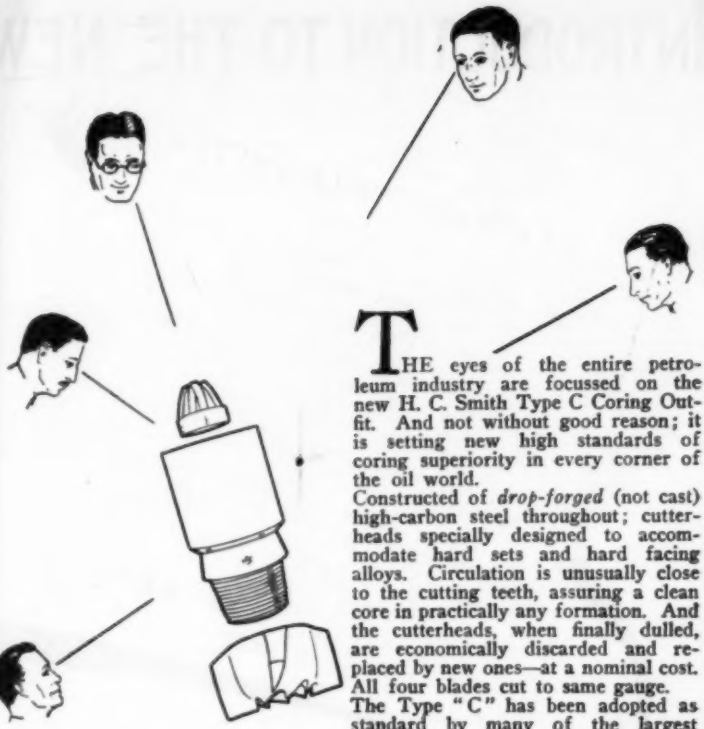

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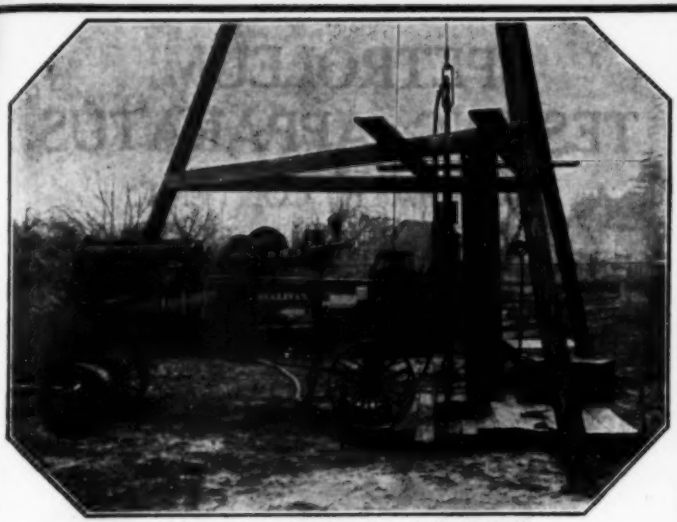

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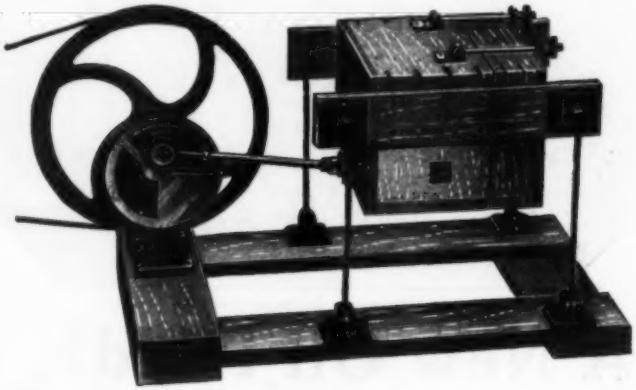

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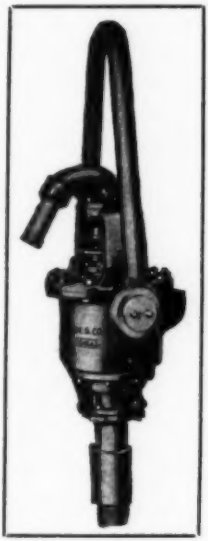

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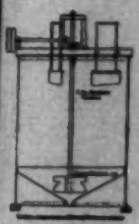

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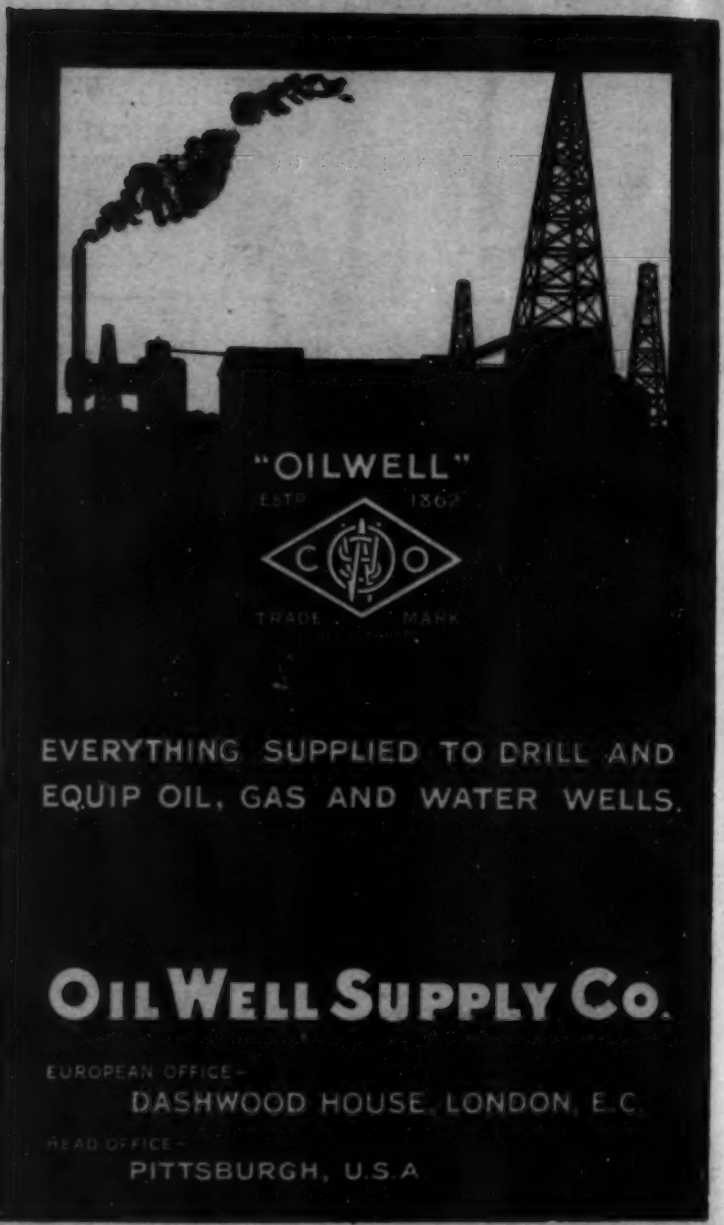

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